SANDIA REPORT

SAND2008-4922
Unlimited Release
Printed August 2008

Supersedes SAND2007-7027
dated October 2007

Long-Term Corrosion Testing Plan

Frederick D. Wall, Neil R. Brown

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

Sandia is a multiprogram laboratory operated by Sandia Corporation,
a Lockheed Martin Company, for the United States Department of Energy's
National Nuclear Security Administration under Contract DE-AC04-94AL85000.

Issued by Sandia National Laboratories, operated for the United States Department of Energy by Sandia Corporation.

Available to DOE and DOE contractors from
U.S. Department of Energy
Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831

Telephone: (865) 576-8401
Facsimile: (865) 576-5728
E-Mail: reports@adonis.osti.gov
Online ordering: http://www.osti.gov/bridge

Available to the public from
U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Rd.
Springfield, VA 22161

Telephone: (800) 553-6847
Facsimile: (703) 605-6900
E-Mail: orders@ntis.fedworld.gov
Online order: http://www.ntis.gov/help/ordermethods.asp?loc=7-4-0#online

SAND2008-4922
Unlimited Release
Printed August 2008

Supersedes SAND2007-7027
dated October 2007

Long-Term Corrosion Testing Plan

Frederick D. Wall
YMP Corrosion Sciences Department
Sandia National Laboratories
P.O. Box 5800
Albuquerque, NM 87185-0888

Neil R. Brown
Geochemical Engineering and Research Group
Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545

Abstract

This document describes the testing and facility requirements to support the Yucca Mountain Project long-term corrosion testing needs. The purpose of this document is to describe a corrosion testing program that will (a) reduce model uncertainty and variability, (b) reduce the reliance upon overly conservative assumptions, and (c) improve model defensibility. Test matrices were developed for 17 topical areas (tasks): each matrix corresponds to a specific test activity that is a subset of the total work performed in a task. A future document will identify which of these activities are considered to be performance confirmation activities. Detailed matrices are provided for FY08, FY09 and FY10 and rough order estimates are provided for FY11-17. Criteria for the selection of appropriate test facilities were developed through a meeting of Lead Lab and DOE personnel on October 16-17, 2007. These criteria were applied to the testing activities and recommendations were made for the facility types appropriate to carry out each activity. The facility requirements for each activity were assessed and activities were identified that can not be performed with currently available facilities. Based on this assessment, a total of approximately 10,000 square feet of facility space is recommended to meet all future testing needs, given that all testing is consolidated to a single location. This report is a revision to SAND2007-7027 to address DOE comments and add a series of tests to address NWTRB recommendations.

CONTENTS

TABLES

ACRONYMS

AES	Auger electron spectroscopy
AFM	atomic force microscopy
BSW	basic saturated water
CDF	cumulative distribution function
CNWRA	Center for Nuclear Waste Regulatory Analysis
CP	cyclic polarization
CT	compact tension
CTL	Corrosion Test Laboratories
CW	cold work
DCPD	direct current potential drop
DI	deionized
DOE	Department of Energy
DSNF	defense spent nuclear fuel
EBS	engineered barrier system
EBSD	electron backscattered diffraction
E_{CORR}	open circuit corrosion potential
EDS	energy dispersive x-ray fluorescence spectroscopy
EIS	electrochemical impedance spectroscopy
E_{RCREV}	crevice repassivation potential
FIB	focused ion beam
FY	fiscal year
GC	general corrosion
GEGRC	General Electric Global Research Center
HAZ	heat affected zone
HIC	hydrogen induced cracking
INL	Idaho National Lab
LC	localized corrosion
LL	Lead Lab
LLNL	Lawrence Livermore National Lab
LPB	low-plasticity burnishing
LPR	linear polarization resistance
LRO	long-range ordering
LTCTF	long term corrosion test facility
m	molal

M	molar
MA	mill annealed
MIC	microbial influenced corrosion
NA	not applicable
RH	relative humidity
SA	solution annealed
SAW	simulated acidified water
SCW	simulated concentrated water
SDW	simulated dilute water
SCC	stress corrosion cracking
SECM	scanning electrochemical microscopy
SEM	scanning electron microscopy
SHT	solution heat treat
SIMS	secondary ion mass spectroscopy
SNL	Sandia National Laboratories
SR	stress relieved
SS	stainless steel
TAD	transportation aging and disposal (waste canister)
TBD	to be determined
TCP	topologically close packed
T_D	dewpoint temperature
TDS	thermal desorption spectroscopy
TEM	transmission electron microscopy
THE	Tsujikawa-Hisamatsu electrochemical (technique)
TOF-SIMS	time-of-flight secondary ion mass spectroscopy
TTT	time-temperature transformation
TWP	technical work plan
WPOB	waste package outer barrier
XPS	x-ray photoelectron spectroscopy
YMP	Yucca Mountain Program

1. INTRODUCTION

This document describes the testing activities planned for FY08 through FY17 for the Yucca Mountain Project Corrosion Testing Program. Detailed test matrices are provided for FY08, FY09 and FY10 with rough order estimates of the testing to be performed in FY11-17. Individual test matrices describe the material types, environments and testing approach necessary to develop the desired information for each activity. The testing details are used to develop facility recommendations for each activity. The criteria for assigning a facility to an activity were derived from a joint meeting between Lead Lab (LL) and Department of Energy (DOE) personnel on October 16-17, 2007. A brief summary of this process, the developed criteria and the recommendations can be found in Section 3 of this document.

1.1 Purpose

The purpose of this document is to provide an overview of the planned testing activities for FY08-FY17 for the Yucca Mountain Corrosion Test Program including recommendations for the type of facility where the work will be performed. This document will provide guidance to testing activities, and individual technical work plans (TWPs) will be developed to provide details of the scientific approach for each activity consistent with the content of this document. A future document will identify which of these activities are considered to be performance confirmation activities.

1.2 Scope

The scope of this document is to provide sufficient detail on planned test activities such that more specific plans for budget, locations and testing resources can be formulated on a year-by-year basis. This document does not constitute a TWP and thus does not contain the information required for a TWP.

1.3 Organization of this document

This document is broken down into three main sections:

1. Introduction
2. Corrosion testing
3. Facilities

The *Corrosion Testing* section contains two sub-sections. The first sub-section, *Scientific Approach*, gives an overview of the materials, environments and processes that are integral to a sound corrosion test program. This sub-section does not provide experimental details, but relays the intent of the testing program. The second sub-section, *Testing Activities*, contains test matrices for activities in fiscal years FY08 through FY17 and the bases for test prioritization. Detailed matrices are provided for FY08, FY09 and FY10 while a rough order estimate is given for FY11-17. These matrices describe the materials, sample configurations, environments and tests or analyses that are needed to meet the goals associated with each activity.

The *Facilities* section describes the process used to develop criteria for facility selection, the developed criteria, the recommended facility assignments for each activity, a gap analysis of required versus available capability, a description and plan for obtaining the additional capabilities needed to support some of the FY11+ testing, and lessons learned from operation of the previous long term corrosion test facility (LTCTF) at Lawrence Livermore National Lab (LLNL).

2. CORROSION TESTING

2.1 Scientific Approach

2.1.1 Materials

Baseline materials—The license application design includes an engineered barrier comprised, in part, of a titanium drip shield and Alloy 22 waste package outer corrosion barrier. Thus, appropriate forms of these construction materials will be the most prominent in the current test plans. Specifically, a range of Alloy 22 compositions will be tested that effectively bound the range of compositions that are expected to be used in the waste package outer barrier (WPOB). Titanium Grade 7 (plate material), Titanium Grade 29 (structural material) and Titanium Grade 28 (weld filler material) will be extensively tested as these are the materials of construction for the drip shield. Borated stainless steel will be tested, as it is the primary neutron absorber material.

Other materials will be included in the test program that do not directly contribute to the barrier function but are materials of construction for support structures and systems. Stainless steel (SS) is used in the rockbolts and perforated ground support structure. The pallet pedestals are Alloy 22 with stainless steel pedestal connectors. Stainless steel is also used in the inner vessel, the transportation aging and disposal (TAD) canisters and the defense spent nuclear fuel (DSNF) canisters. Carbon steel and crushed tuff ballast is used in the invert as structural support to waste packages and emplacement equipment. Interactions between titanium and Alloy 22 with these various materials will be part of the investigations.

Analogues and less resistant materials—Determining how contributing factors such as composition, metallurgy and environment contribute to a given damage mechanism, damage rate, and total accumulated damage is difficult if only baseline materials are tested. Alloy 22 and titanium have such low rates of corrosion and such high resistance to localized corrosion processes that differentiating the relationships between environmental stresses and material responses can only be achieved with limited fidelity using traditional testing approaches. By including materials in the test program that have less corrosion resistance it is possible to build knowledge of fundamental relationships between material characteristics and corrosion mechanisms. These relationships can then be used to make predictions or improve confidence in predictions of the baseline material behavior.

Less corrosion resistant analogues tested in parallel with the baseline materials also help to define the margins of behavior for the baseline materials. For instance, if it can be demonstrated that a less corrosion resistant nickel-chromium-molybdenum alloy such as C276 has sufficient corrosion resistance to meet repository needs then little doubt would exist as to the reliability of Alloy 22.

Alternative/replacement materials—The waste package design may not remain static throughout the period of repository operation: new materials may be incorporated into the design or current materials that do not receive barrier function credit may be re-evaluated. The long term corrosion science program must be proactive in anticipating changes to barrier design or barrier credit and carry out a test program that includes these elements as resources allow.

Many materials that serve as analogues to Alloy 22 may also be considered as alternative barrier materials, so testing of this class of materials will benefit Yucca Mountain Project in multiple areas. Additionally, new materials such as rapidly solidified iron-based coatings could be included in portions of the test matrix to ensure the Project can take advantage of emerging technologies. Other novel materials may be added to the testing program as they are developed.

The class of materials used for the inner vessel of the waste package will also be included in the corrosion test program. The current waste package design includes a 5 cm thick inner vessel constructed of 316 SS. No waste containment barrier credit is given to this material but enhanced understanding and better predictive models may allow the project to take credit for this vessel in future licensing efforts. Alternative materials to 316 SS should also be considered, including other stainless steels and Ni-based alloys. Materials used to construct the shell of the TAD will also be represented in the test program consistent with available resources.

Material condition— The condition of a material is, in many cases, just as critical as the composition in determining corrosion resistance. The waste package will be fabricated from mill annealed (MA) sheets that are welded together. After welding, the Alloy 22 will be solution annealed (SA) and quenched and the resulting black annealing film will be removed. Testing will be performed both with and without removal of the black anneal film to provide possible processing options to future engineering efforts. For safety and practical reasons, the outer closure weld, however, will not be solution annealed but will be stress mitigated with low plasticity burnishing (LPB). Therefore, the welded and solution annealed condition is of primary interest for the corrosion of Alloy 22, with analysis required to support modeling of the welded and stress mitigated outer closure weld region, particularly with regards to resistance to localized corrosion (LC) and stress corrosion cracking (SCC). The drip shield will be fabricated from Titanium Grade 7 plates with Titanium Grade 29 structural material. The Titanium Grade 7 to Grade 29 welds will be performed with Titanium Grade 28 weld filler material. After welding, the drip shield will be stress relief annealed. Therefore, the welded and heat treated condition is of primary interest for the corrosion of titanium.

The surface roughness of exposed materials is also important in determining the likelihood that LC will initiate. Testing will include expected ranges of surface finishes, especially for crevice corrosion and stress corrosion cracking SCC initiation. Surface finish is also affected by normal and abnormal handling and emplacement.

2.1.2 Environments

The emplaced waste packages and drip shields will experience a wide range of environments during the period of repository performance. The nominal environment at any given time step will be defined by the prevailing temperature, humidity and seepage conditions. The ionic makeup of any electrolyte present will be largely determined by constituents of the dust that settles on metal and the seepage waters from the host rock. Local environments may develop at sites of metal-metal contact (waste package contacting pallet or collapsed drip shield contacting the waste package), under delaminated oxides, under rubble due to drift collapse or backfill, under precipitated salt films or at sites of microbial activity. A comprehensive test program will include facilities to reproduce the variety of expected environments and appropriate experimental techniques to appropriately characterize material behavior in each of the environments.

Seepage conditions—Once the drift wall temperature drops below boiling, seepage environments are expected to form then remain for the duration of the repository performance period. Thus this class of environments, defined by seepage in contact with the engineered materials, is expected for up to hundreds of thousands of years. Although the temperature is only moderate under seepage conditions, the long time period and possibility of aggressive electrolytes makes this environment critical to repository performance.

To date the effects of direct seepage contact have been estimated by testing samples completely immersed in aqueous electrolyte. Despite physical differences between the true seepage condition and the experimental condition, this approach allows many environmental parameters of temperature and chemistry to be explored and well-established corrosion testing techniques to be applied. Immersion testing will continue to be a significant effort in the ongoing experimental program. A range of anticipated temperatures and electrolyte compositions can be studied. Inundated experiments are conducive to long-term testing as maintaining the environment is straightforward and many different sample configurations can be simultaneously tested.

Much of the engineered material surface area will not be directly or continuously contacted by seepage in the repository but will be exposed to humidity, temperature and any contaminants from either dust, prior seepage events or gas phase contaminants. Samples in the LTCTF were suspended above the electrolyte phase in order to simulate atmospheric corrosion conditions. A more rigorous approach is to study samples in flow-through atmospheric test chambers where temperature, relative humidity (RH) and gas composition is carefully controlled. Samples can be loaded with contaminants through particle deposition schemes, exposure to simulated dripping conditions and through other preconditioning approaches. Predicted gas phase composition from repository environmental models can be used to determine the appropriate environments for the test program. The gas phase can be controlled through the use of permeation tubes, gas cylinders and appropriate mixing with mass-flow controllers.

A methodology for simulating dripping conditions will be developed to complete the experimental approach to evaluating effects of seepage conditions. Processes such as salt separation (a process where minerals systematically precipitate as a function of equilibrium RH and solution concentration) cannot be directly studied without the means of reproducing the associated environment in the laboratory. As a result, the models use very conservative treatments for salt separation conditions. Additionally, complex and coupled processes such as contaminant supply and participating cathodic area are directly related to the physics of the dripping process. Corrosion scientists and geochemists will define and implement a simulated seepage testing capability.

Deliquescent conditions—The deliquescent period of concern lasts a relatively short period of time (i.e., thousands and not tens of thousands of years) and is defined by conditions where the temperature and water saturation is such that multi-salt assemblages that could conceivably exist in dust deposits may deliquesce on the waste package surface at temperatures higher than seepage conditions. The electrochemical environments associated with deliquescence are generally characterized in terms of salt composition and ratio of nitrate to chloride. Nevertheless, the very high temperatures, up to approximately 200°C, where deliquescence can occur raises the concern that localized corrosion or exacerbated general corrosion could occur

and that such damage modes cannot be predicted by extrapolation of results from testing at lower temperatures under inundated conditions in less concentrated brines.

Three general approaches to simulating deliquescence conditions have been taken by the project thus far: (1) testing in concentrated brine solutions (at atmospheric pressure), (2) testing in concentrated brine solutions within autoclaves at elevated temperature and pressure, and (3) testing in high-temperature steam. Testing in concentrated brines is appropriate for exploring the effects of anion concentration on LC initiation and for determining basic corrosion parameters such as open circuit corrosion potential (E_{CORR}) and crevice repassivation potential (E_{RCREV}). However, results from such testing should not be used in the absence of other information. The concentrated brine solutions do not represent the condition of a thin or discontinuous electrolyte nor the limited quantity of reactants that will actually comprise the repository environment. Testing under autoclave conditions is inappropriate as the high pressure and refluxing conditions may allow the environment to evolve to an unknown and potentially unrealistically aggressive state. Testing samples contaminated with particulates in high temperature steam is a good starting point for assessing corrosion under deliquescent conditions. However, additional confidence in behavior can be gained by providing for control over contaminant composition and concentration as well as gas phase composition. Work in this area is already underway and will provide the basis for longer-term testing efforts.

Coupled deliquescent and seepage environments—The possibility exists that sequential exposure to deliquescent environments followed by seepage environments may cause damage beyond decoupled exposures. The most likely mechanism for such a damage accumulation process is that oxide structure and/or composition is modified under deliquescent conditions such that it behaves differently under seepage conditions than predicted by models based on data from MA, welded or thermally aged samples. Although the behavior may be transient in nature until the oxide reaches a new steady-state condition and the behavior may not result in exacerbated damage, a test program of preconditioning samples under deliquescent conditions followed by testing under seepage conditions will fully explore this scenario.

Rubble environments—The waste package or drip shield may come into contact with rubble from the host rock following a seismic event. Additionally, if the repository were to be backfilled (not currently planned) with indigenous rock, a rubble-contact environment would exist from the moment of emplacement. Neither of these scenarios has been the subject of in depth study in the corrosion test program. The chemical and physical effects of rubble contact will be studied in the next phase of corrosion research.

The chemical effects of rubble contact can be assessed to a degree by performing some of the conventional tests under appropriately modified environments. The geochemical models of the Yucca Mountain site and in-drift conditions should be used to establish bounds on the expected chemical environments.

The physical effects of rubble contact will be to alter the extent and footprint of electrolyte contact and to form crevices where there is direct physical contact. The more critical of these two factors is believed to be formation of crevices. A test program to evaluate the parameters that govern the rock-metal crevice behavior will be undertaken. The results of this study will be used to guide corrosion studies with artificial rock-metal crevices.

Microbial influenced corrosion (MIC)—Microbial colonies may establish local environments that differ significantly from the nominal waste package environment. Two lines of investigation are appropriate for further characterization of MIC effects: (1) continuing and extending the previous Yucca Mountain Project work on this topic to evaluate the propensity for a microbial colony to form and to persist, and (2) performing studies to assess the behavior of the engineered materials in the environments that could form due to the presence of a microbial colony, if colonies are believed to be viable.

A survey will be made of environments known to form under biofilms and a subset of these environments will be identified that are relevant to engineered barrier system (EBS) component conditions. From this study a range of environmental parameters will be identified for studying both the general and localized corrosion behavior of the engineered materials. A result of this approach will be the ability to answer the question of what impact microbial colonies could realistically have on repository performance. This information can be combined with the assessment of likelihood of microbial activity to estimate the total contribution of MIC to repository degradation.

2.1.3 Processes and Mechanisms

A comprehensive assessment of the reliability of the engineered materials in the repository can be made by considering potential corrosion modes and confirming the anticipated behavior through a combination of experimentation and modeling. To date, the experimental program has made efforts to characterize the primary corrosion modes: general corrosion, localized corrosion and environmental cracking including hydrogen induced cracking (HIC). The emphasis has been on measuring rates, critical values of potential and stress intensity and developing associated empirical models. These are valuable approaches and go a long way towards establishing confidence in repository safety, but as these test programs are continued and expanded, it is imperative that increasing resources be applied to developing fundamental understanding, characterizing processes at appropriate length scales, and building a close relationship between modeling and experimental activities. Such an understanding will increase the confidence in the existing models and reduce the reliance upon conservative but unrealistic modeling assumptions.

Oxide properties—The thin oxide layers on titanium alloys, nickel alloys and stainless steels determine the passive corrosion rates in most environments, dictate the localized corrosion behavior, and contribute significantly to the stress-corrosion cracking behavior. An experimental program to predict the corrosion behavior of passive metals over the regulatory period of the repository will include comprehensive tasks to characterize oxide structure and composition, develop understanding of the impact of environmental stresses on oxide stability, and characterize growth and repassivation behavior.

The oxide structure and composition will be determined on the smallest relevant length scale that is possible. Increased confidence in prediction of corrosion processes will emerge from being able to quantify oxide behavior at the nanoscale. Characterization techniques such as small spot size x-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS), and Auger electron spectroscopy (AES) will help to determine oxide chemistry. Atomic force microscopy (AFM), cross-sectional transmission electron microscopy (TEM) and field emission scanning electron microscopy (SEM) will contribute toward an understanding of oxide structure

and topography. Only by having a thorough understanding of the starting oxide condition is it possible to track subtle changes due to exposure to the environment. Importantly, this effort should include characterization of oxide repassivation behavior as this could be a controlling factor in the overall alloy stability.

Imperative to correctly characterizing and modeling all corrosion modes is knowledge of the oxide evolution during the repository performance period. Thus short and long-term corrosion studies will be integrated with the oxide characterization task. Issues such as coupled deliquescent and seepage processes are directly dependent on the evolution of the oxide properties. Corrosion studies will iteratively provide inputs to and receive information from the oxide characterization work. As an example, investigation of general corrosion rate may yield information on oxide thickening and annealing which in turn will contribute to oxide electronic and ionic conduction properties. The evolution of these properties will be characterized and used as inputs to the localized corrosion studies.

Aging and phase stability—Of key importance to all of the corrosion modes is the composition and microstructure of the material under investigation. Thermal treatments can alter the local microstructure through phase precipitation and ordering. Repository materials will be subject to processes such as welding, annealing, quenching and aging during the thermal period of the repository. It is important to build an understanding of the range of microstructures that may exist in the repository and the implications for anticipated corrosion processes. A particularly useful approach that has been used in other alloy systems is to catalogue the behavior of individual phases or solute depleted regions that form due to thermal aging. A potential approach to this type of study is to utilize micro-capillary electrochemical techniques or surface probe techniques such as electrochemical AFM or scanning electrochemical microscopy (SECM).

General corrosion—General corrosion (GC) behavior of the engineered materials in the repository is a continuous and inevitable process. The primary questions concern rate, environmental dependence, spatial variability and uncertainty. Testing to date has mostly been performed under inundated conditions in a range of electrolytes and temperatures. Measurement techniques have included short term electrochemical measurements and longer term weight loss measurements. Due to the excellent corrosion resistance of Alloy 22 and titanium, the influences of electrolyte composition have not been conclusively determined resulting in the conservative treatment that the GC rate is independent of environment. A temperature dependence has been established for Alloy 22 with a large range of activation energies, but none has been established for titanium.

Work will continue to characterize GC behavior under realistic repository environments. While inundated testing may provide an upper bound for some of the anticipated seepage conditions, it would be advantageous to improve understanding of GC under atmospheric, dripping and deliquescent conditions. Test techniques will be developed to provide a semi-quantitative understanding of the relationship of corrosion rates under inundated conditions and those in humid air.

The general corrosion rate is observed to decrease with exposure time for many metal-electrolyte systems. Therefore, more accurate indications of long-term behavior are gained by increasing

exposure times. The test plan for general corrosion behavior will have, at its core, a long-term exposure program where coupons are immersed under a variety of environmental conditions for tens of years. Samples will be included that are periodically monitored with electrochemical techniques such as electrochemical impedance spectroscopy (EIS) or linear polarization resistance (LPR). Samples will also be included that are characterized with weight loss measurements – here, it is important to select samples optimized for measurement sensitivity (e.g., high surface area to volume ratio) balanced against the need to ensure that samples are robust enough for cleaning and weighing. Additional samples will be tested in parallel that are characterized with surface analytical techniques, surface probes and cross sectional SEM and TEM. The Project baseline general corrosion model is currently based upon dissolution rate under open circuit conditions. The material in the repository may encounter oxidizing environments due to Fe ions from corroded structures, due to hydrolysis from radiation or due to contact with a dissimilar metal. The experimental work in support of the GC model will investigate the passive corrosion rate of Alloy 22 at applied potentials above the open circuit potential (possibly through the use of galvanic couples) and/or in oxidizing environments to address these issues.

Material metallurgy has a significant effect on GC behavior. The corrosion program will include testing to determine the impact of alloy composition, weld regions, and microstructure evolution due to thermal aging effects, and stress mitigation.

Crevice corrosion—The model for crevice corrosion conservatively relies on a criteria for initiation without the possibility of stifling or arrest mitigating the extent of accumulated damage. To date, most of the experimental work performed on the Project has focused on measuring parameters related to predicting initiation. Other work on initiation has been in the form of inspecting crevice coupons after long-term exposure to repository relevant and accelerating environments. Crevice propagation has been conservatively estimated as the average dissolution rate of Alloy 22 in highly aggressive acid solutions at elevated temperature. The result of this work is a conservative estimation of the waste package resistance to crevice corrosion. A comprehensive understanding of all phases of crevice corrosion would improve confidence in the existing model and might enable the use of a less conservative model.

In the current baseline model, crevice initiation is assumed to occur when the sample E_{CORR} exceeds E_{RCREV} for a particular environment. The use of E_{CORR} in this prediction carries an implicit assumption that the E_{CORR} value measured under free corrosion conditions can be maintained when a crevice begins to draw cathodic current. This assumption is overly conservative and will lead to unrealistic predictions of crevice corrosion behavior. To improve the description of crevice initiation, the cathodic kinetics on Alloy 22 will be determined as a function of potential and environment and the cathodic kinetics correlated to the oxide structure and composition.

The scope of the experimental studies on crevice corrosion will be expanded to produce detailed understanding of the propagation, stifling and arrest phases. The likelihood that a crevice, once initiated under seepage conditions, will be sustained such that breach of a barrier occurs cannot be estimated with current Project data. It is also unknown how a repassivated crevice will respond to environmental stresses. Therefore, the project has assumed that once localized corrosion is initiated it will continue until waste package penetration. The actual degradation

process will likely be long and complex with failure the result of many coupled corrosion processes rather than one defining event such as a single instance of crevice initiation.

In the baseline model, crevice corrosion is predicted to occur any time an electrochemical criterion, $E_{CORR} > E_{RCREV}$, is met independent of the surface condition of the material and independent of the physical configuration of the crevice. An experimental effort can help determine how the surface condition of the barrier material impacts the propensity for crevice initiation. This includes studies of surface roughness, defects due to manufacture and emplacement, defects due to seismic activity, and the presence of surface scales from annealing or salt and mineral precipitation. Crevice corrosion will also be highly dependent on the type of crevice former (e.g., metal, rock, or scale), the physical condition of the crevice former (e.g., porosity) and the tightness and dimensions of the crevice. These parameters will impact crevice stifling and arrest as well as initiation and the experimental program should cover all stages of crevice corrosion.

The physical and chemical nature of the waste package environment will impact all stages of crevice corrosion. For instance, under deliquescent environments the connectivity of electrolyte may be highly discontinuous, thus limiting the cathodic area that can support a particular crevice site. Additionally, limits on available quantities of reactants may result in crevice stifling or arrest. Under seepage conditions the local environment may have a limited delivery rate or limited total quantity of electrolyte that limits the available reactants and cathodic area. Chemical effects also include the presence of inhibiting species such as nitrate, oxidizing species such as ferric ion or peroxide, and aggressive halide ions such as chloride or fluoride. Additional confidence in the corrosion modeling can be gained by taking these factors into account and developing a mechanistic description of the crevice corrosion process that spans from initiation to arrest, through multiple cycles of active corrosion and passive behavior.

Pitting and metastable pitting—The LC studies performed on the project to date have focused on crevice corrosion with the assumption that crevice corrosion can be used to conservatively represent all forms of localized corrosion. However, it should be noted that metastable pitting processes may occur at potentials below where stable pitting is observed. Over the lifetime of the repository damage may accumulate from processes that taken in isolation do not represent a classical threat to material integrity. A complete corrosion program for Yucca Mountain Project will include an effort to characterize metastable pit processes on passive materials and an effort to understand the factors that can sustain a transition to stable pit growth.

Investigating pitting behavior in highly passive materials such as titanium and Alloy 22 requires that the experimental work be focused at a length scale where relevant processes can be adequately characterized. Oxide modification including defect incorporation, thinning, and pore formation will be studied at the nanometer length scale.

Stress Corrosion Cracking—SCC is modeled to initiate in the engineered barrier materials when a critical stress threshold is exceeded on smooth surfaces or when a critical stress intensity is exceeded on welded surfaces that contain weld flaws. Crack growth is assumed to occur when the threshold stress intensity value is exceeded and crack growth velocity is represented as a function of the stress intensity at the crack tip. Importantly, the cracking criteria do not include

environmental effects (e.g. temperature or solution chemistry thresholds) or metallurgical condition, which would act to reduce the possibility of SCC.

Understanding the metallurgical or environmental contributions to cracking in a highly resistant material is difficult because the resolution needed to appropriately quantify and differentiate rates as a function of these parameters is not straightforward to achieve. Tests either have to be run for extended lengths of time or the measurement of crack length has to be sufficiently sensitive. In order to overcome this barrier, part of the long-term effort to characterize SCC behavior should focus on improving the measurement techniques and developing better long-term testing approaches that can provide differentiating, quantitative information.

The SCC susceptibility of Alloy 22 and titanium can be understood in terms of the three contributions to this failure mode: microstructure, environment, and stress intensity. Microstructure effects may include phase formation, solute segregation/depletion and long-range ordering. Important environmental effects include halide ion concentration, inhibiting ion concentration, pH and temperature.

The use of a film rupture slip dissolution model to estimate crack velocity for the engineered barrier materials requires estimation of the oxide repassivation kinetics and the development of relationships governing the calculation of various constants in the model. To date much of this information has been estimated by using values from 304 SS in 288ºC water. As resources allow, future experimental efforts will establish the model parameters for the engineered barrier materials in repository relevant environments. Fundamental understanding of oxide breakdown and growth processes will also be integral to using and validating the film rupture slip dissolution model.

Hydrogen Induced Cracking—Currently, the hydrogen content of Titanium alloys in the repository is estimated from the general corrosion rate (measured via weight loss) and an assumed value for the absorption efficiency. Failure from HIC is screened out based upon modeling predictions because the calculated hydrogen concentration does not exceed a threshold value. In the case of Alloy 22, HIC is screened out based on the high resistance to HIC of other nickel-chromium-molybdenum alloys in the annealed condition.

A more quantitative understanding of HIC effects will be developed for all barrier materials in the repository. Part of this understanding will come from assessing the actual levels of hydrogen that must be achieved to have a detrimental impact on mechanical behavior. The relationship between corrosion processes and hydrogen uptake will also be established for the barrier materials. Using an approach like thermal desorption spectroscopy in conjunction with electrochemical testing will enable determination of the trap sites, binding energies and migration energies for hydrogen in these materials.

2.2 Testing Activities

Testing activities are broken down into 17 tasks to account for the various types of tests and materials. Each of these matrices in turn is broken down into detailed test matrices for FY08, FY09 and FY10, with rough order of magnitude estimates for FY11 through FY17.

The testing identified for FY08 is designed to (a) improve confidence in predicting long-term corrosion behavior through analyses of the 9.5 year immersion samples, and oxide film characterization, (b) reduce uncertainty in current models be replicating tests with large data variability and/or outliers, (c) perform tests to demonstrate corrosion stifling under dust deliquescence conditions, and (d) continue ongoing testing to support a future screening of stress corrosion cracking.

Testing in FY09 and FY10 focuses primarily upon a systematic investigation of the underlying corrosion processes to inform performance confirmation, performance margin analysis and address secondary issues.

Testing in FY11 through FY17 will focus on long-term tests and secondary issues. The matrices provided for FY11 through FY17 represent a best estimate at this time and are likely to change substantially as a result of information gathered in FY08 through FY10. These matrices provide sufficient detail for a bottoms up estimate of facility capability, sample and equipment needs.

The 17 tasks described in this document are defined as follows:

Task 1: Weight Loss for Alloy 22 and Titanium and Surrogates
Task 2: Crevice Corrosion of Alloy 22, Titanium Alloys and Surrogates
Task 3: Critical Potential of Alloy 22
Task 4: Open Circuit Potential of Alloy 22
Task 5: Temperature Dependence of Alloy 22 and Titanium Alloys
Task 6: Critical Potential of Titanium Alloys
Task 7: Characterization of Alloy 22 and Titanium Oxides
Task 8: Alloy 22, Deliquescence Testing
Task 9: Microbial Influenced Corrosion on Alloy 22
Task 10: Aging and Phase Stability of Alloy 22
Task 11: Stress Corrosion Cracking
Task 12: Analysis of U-bend Samples from the LTCTF
Task 13: Hydrogen Embrittlement
Task 14: Corrosion Behavior of Neutron Absorber Materials
Task 15: Analysis of Stainless Steel Corrosion Products
Task 16: Corrosion Testing Under Dripping Conditions
Task 17: Open Circuit Potential of Titanium

2.2.1 Task 1: Weight Loss for Alloy 22 and Titanium and Surrogates

The objective of this task is to improve confidence in the general corrosion rate models and predictions; improve understanding of the influence of temperature, chemical environment and exposure time on corrosion rate; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for general corrosion behavior of Alloy 22 and titanium alloys described, respectively, in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS180778]) and *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

Archived samples that were exposed in the LTCTF will be analyzed and documented for weight loss and physical appearance according to the matrices provided in Tables 2.2.1.1 through 2.2.1.3. The analyses will include optical microscopy of the sample surfaces prior to and following cleaning to remove corrosion products and weighing of the samples after removal of corrosion products. Select samples will be imaged using SEM as appropriate. One sample will be held out from the welded/crevice category for each environmental condition (a total of 12 coupons will be reserved). These samples will be archived for surface analysis. A sample will also be held out from the welded/non-crevice category for each environmental condition. These samples will be used for weight loss if it is deemed that an additional data point is necessary for improving the weight loss statistics . Otherwise these samples will be archived for future analysis.

Long-term exposure testing will be performed on the baseline materials for the waste package outer barrier including Alloy 22, Titanium Grade 7 and Titanium Grade 29 (Table 2.2.1.4). The environments were selected to evaluate the effects on general corrosion rate of the anticipated primary variables of temperature, chloride concentration and nitrate concentration and the anticipated secondary variables of lead, fluoride, bicarbonate and silicate. The selection of to-be-determined (TBD) environments will be based primarily on results and analyses from testing performed during FY08 and FY09. The listed environments may also be modified if further analysis indicates that a different selection of environments, but the same approximate total number, would provide more complete information or information that better targets a specific corrosion concern.

Long term exposure testing will also be performed on thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (Table 2.2.1.5). These tests will determine any differences between the behavior observed for baseline materials (Table 2.2.1.4) and other mechanical or thermal conditions, other pretreatments (e.g., black anneal or deliquescent pre-exposure) and galvanic coupling. These tests will also provide information on how lower alloyed nickel-chromium-molybdenum materials behave compared to Alloy 22 and how potential surrogate titanium alloys behave compared to Titanium Grade 7 and Titanium Grade 29.

Table 2.2.1.1. Task 1, FY08 Matrix 1: Weight loss and crevice specimens to be photographed and weighed. The FY08 matrix applies to Alloy 22 (9.5 years exposure), Titanium Grade 7 (6.6 years exposure) and Titanium Grade 16 (9.5 years exposure).

Electrolyte	Phase	Fabrication	Temperature			
			60°C		90°C	
SDW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SAW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SCW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3

Table 2.2.1.2. Task 1, FY09 Matrix 1: Weight loss and crevice specimens to be photographed and weighed. The FY09 matrix applies to Alloy 625 (9.5 years exposure) and Alloy C-4 (9.5 years exposure).

Electrolyte	Phase	Fabrication	Temperature			
			60°C		90°C	
SDW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SAW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SCW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3

Table 2.2.1.3. Task 1, FY10 Matrix 1: Weight loss and crevice specimens to be photographed and weighed. The FY10 matrix applies to Alloy G3 (9.5 years exposure) and Alloy 825 (9.5 years exposure).

Electrolyte	Phase	Fabrication	Temperature			
			60°C		90°C	
SDW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SAW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
SCW	Aqueous		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3
	Vapor		Weight Loss	Crevice	Weight Loss	Crevice
		Weld	2 or 3	2	2 or 3	2
		No Weld	3	3	3	3

Table 2.2.1.4. Task 1, FY11-17 Matrix 1: Rough order estimate of long-term weight-loss coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29 (6,606 samples, including TBDs).

$[Cl^-]$ / *m*	$[NO_3^-]$ / *m*	Other composition	T (°C)	# Samples of each material analyzed at each time period								
				0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
1	0	-	30	6	6	6	6	6	6	10	10	10
1	0	-	60	6	6	6	6	6	6	10	10	10
1	0	-	90	6	6	6	6	6	6	10	10	10
1	0	-	98	6	6	6	6	6	6	10	10	10
0.1	0	-	90	6	6	6	6	6	6	10	10	10
10	0	-	60	6	6	6	6	6	6	10	10	10
1	0.01	-	30	6	6	6	6	6	6	10	10	10
1	0.1	-	60	6	6	6	6	6	6	10	10	10
1	1	-	90	6	6	6	6	6	6	10	10	10
-	-	SAW	30	-	-	-	6	6	6	10	10	10
-	-	SAW	60	-	-	-	6	6	6	10	10	10
-	-	SAW	90	-	-	-	6	6	6	10	10	10

[Cl^-] / *m*	[NO_3^-] / *m*	Other composition	T (°C)	# Samples of each material analyzed at each time period								
				0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
-	-	SDW	30	-	-	-	6	6	6	10	10	10
-	-	SDW	60	-	-	-	6	6	6	10	10	10
-	-	SDW	90	-	-	-	6	6	6	10	10	10
-	-	SCW	30	6	6	6	6	6	6	10	10	10
-	-	SCW	60	6	6	6	6	6	6	10	10	10
-	-	SCW	90	6	6	6	6	6	6	10	10	10
0	0	Distilled Water	90	6	6	6	6	6	6	10	10	10
1	0	1 ppm Pb	90	-	-	-	6	6	6	10	10	10
1	0	10 ppm Pb	90	-	-	-	6	6	6	10	10	10
1	0.1	10 ppm Pb	90	-	-	-	6	6	6	10	10	10
1	0	0.02 *m* F	90	-	-	-	6	6	6	10	10	10
1	0	0.2 *m* F	90	-	-	-	6	6	6	10	10	10
1	0.1	0.2 *m* F	90	-	-	-	6	6	6	10	10	10
1	0	0.02 *m* HCO_3^-	90	-	-	-	6	6	6	10	10	10
1	0	0.2 *m* HCO_3^-	90	-	-	-	6	6	6	10	10	10
1	0.1	0.2 *m* HCO_3^-	90	-	-	-	6	6	6	10	10	10
1	0	4 mg/l SiO_3^{2-}	90	-	-	-	6	6	6	10	10	10
1	0	40 mg/l SiO_3^{2-}	90	-	-	-	6	6	6	10	10	10
1	0.1	40 mg/l SiO_3^{2-}	90	-	-	-	6	6	6	10	10	10
-	-	TBD-1	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-2	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-3	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-4	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-5	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-6	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-7	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-8	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-9	-	TBD	TBD	TBD	6	6	6	10	10	10
-	-	TBD-10	-	TBD	TBD	TBD	6	6	6	10	10	10

Table 2.2.1.5. Task 1, FY11-17 Matrix 2: Rough order estimate of long-term weight-loss coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (6340 samples, including TBDs).

Environments: **1 *m* NaCl, 90°C, neutral pH** **1 *m* NaCl + 0.1 *m* $NaNO_3$, 90°C, neutral pH** **SAW, 90°C** **SCW, 90°C**										
		# Samples analyzed at each time period, per environment, per material								
Material	**Condition**	**0.1 yr**	**0.25 yr**	**0.5 yr**	**1 yr**	**2 yr**	**5 yr**	**10 yr**	**20 yr**	**Extra**
Alloy 22	Black Anneal[1]	60	60	60	60	60	60	60	60	10
Alloy 22	LPB	-	-	6	6	6	6	10	10	10
Alloy 22	Simulated weld root	-	-	6	6	6	6	10	10	10
Alloy 22	Simulated HAZ	-	-	6	6	6	6	10	10	10
Alloy 22	Galvanic couple 316SS	-	-	6	6	6	6	10	10	10
Alloy 22	Galvanic couple Titanium Gr. 7	-	-	6	6	6	6	10	10	10
Alloy 22	Deliquescent pre-exposure	-	-	6	6	6	6	10	10	10
C276	Annealed	-	-	6	6	6	6	10	10	10
Ni	Annealed	-	-	6	6	6	6	10	10	10
Nichrome 80-20	Annealed	-	-	6	6	6	6	10	10	10
Ni-20 Cr-7 Mo	Annealed	-	-	6	6	6	6	10	10	10
Ni-15 Cr-7 Mo	Annealed	-	-	6	6	6	6	10	10	10
316 SS	Annealed	-	-	6	6	6	6	10	10	10
Future material	TBD	-	-	6	6	6	6	10	10	10
Titanium Gr7/28/29	Weld	-	-	6	6	6	6	10	10	10
Titanium Grade 28	Annealed	-	-	6	6	6	6	10	10	10
TBD-1	-	-	-	6	6	6	6	10	10	10
TBD-2	-	-	-	6	6	6	6	10	10	10
TBD-3	-	-	-	6	6	6	6	10	10	10
TBD-4	-	-	-	6	6	6	6	10	10	10
TBD-5	-	-	-	6	6	6	6	10	10	10

[1] The black anneal condition will experience mass loss during the cleaning process due to removal of the thermal oxide. Therefore, it is necessary to establish a cumulative distribution function (CDF) for mass-change that represents cleaning of samples that have not been exposed to a test environment. Comparing CDFs from exposed populations of samples will enable a determination as to the effect of black anneal on general corrosion rate. The CDF of untested black anneal Alloy 22 will require 60 samples to have the same data density as the CDFs developed for the various exposure times.

2.2.2 Task 2: Crevice Corrosion of Alloy 22, Titanium Alloys and Surrogates

The objective of this task is to improve confidence in the crevice corrosion initiation models and predictions; improve understanding of the influence of physical crevice parameters (e.g. force, crevice material), chemical environment and exposure time on crevice initiation and damage accumulation; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for localized corrosion behavior of Alloy 22 and titanium alloys described, respectively, in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]) and *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

The archived crevice geometry samples from the LTCTF will be analyzed and documented for physical appearance including evidence of localized corrosion (Table 2.2.2.1). The analyses will include optical microscopy of the sample surfaces prior to and following cleaning to remove corrosion products and select samples will be imaged using SEM as appropriate. One welded sample will be held out for each environmental condition (a total of 12 coupons will be reserved). These samples will be archived for surface analysis. If crevice corrosion is observed or if the initial examination is inconclusive, metallographic cross sectioning may be conducted.

Crevice specimens of Alloy 22 surrogate materials exposed in the LTCTF will be examined in FY09 in order to identify and prioritize specimens for more detailed examination (Table 2.2.2.2). It is assumed that if crevice corrosion has not occurred on these samples that crevice corrosion at earlier times or lower temperatures is unlikely. Priority will be determined by considering the extent of attack and the relevance of the material to Alloy 22. Samples that did not undergo crevice attack will be given low priority. Samples that underwent attack and are compositionally similar to Alloy 22 will be given higher priority. The results of this prioritization will define the specimens to be examined in FY09 (Table 2.2.2.3) and FY10 (Table 2.2.2.4).

Scoping experiments will be performed in FY10 to evaluate damage accumulation due to crevice corrosion on Alloy 22 in selected environments (Table 2.2.2.5). These data will provide guidance to FY11-FY17 activities that investigate the influence of crevice material, electrolyte (including nitrate concentration), crevice force and thermal and mechanical processing effects. These data will also provide the foundational information for constructing a conceptual understanding of damage propagation.

Long-term open circuit exposures will be used to evaluate crevice corrosion behavior of the baseline materials for the waste package outer barrier including Alloy 22, Titanium Grade 7 and Titanium Grade 29 (Table 2.2.2.6). The environments were selected to evaluate the effects on crevice corrosion of the anticipated primary variables of temperature, chloride concentration and nitrate concentration and the anticipated secondary variables of lead, fluoride, bicarbonate and silicate. The selection of TBD environments will be based primarily on results and analyses from testing performed during FY08 and FY09. The listed environments may also be modified

if further analysis indicates that a different selection of environments, but the same approximate total number, would provide more complete information or information that better targets a specific corrosion concern.

Long-term open circuit exposures will also be used to evaluate the crevice corrosion behavior of thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (Table 2.2.2.7). These tests will determine if there are significant differences in behavior between the baseline materials (Table 2.2.2.6) and other mechanical or thermal conditions, other pretreatments (e.g., black anneal or deliquescent pre-exposure) and galvanic coupling. These tests will also provide information on how lower alloyed nickel-chromium-molybdenum materials behave compared to Alloy 22 and how potential surrogate titanium alloys behave compared to Titanium Grade 7 and Titanium Grade 29.

A series of open circuit exposures will be performed on creviced specimens of Alloy 22 to determine the influence of chemical environment, physical environment (e.g., rubble), processing, crevice torque and crevice material on likelihood and extent of crevice propagation (Table 2.2.2.8). These tests will typically be long-term in duration (i.e., 5 years) and will involve removal and analysis of samples at intermediate time steps to track the progression, if any, of localized attack.

Table 2.2.2.1. Task 2, FY08 Matrix 1: Crevice specimens to be photographed and examined for signs of localized corrosion attack. The FY08 matrix applies to Alloy 22 (9.5 years exposure), Titanium Grade 7 (6.6 years exposure) and Titanium Grade 16 (9.5 years exposure).

			Temperature	
Electrolyte	**Phase**	**Weld / No Weld**	**60°C**	**90°C**
SDW	**Aqueous**	Weld	2	2
		No Weld	3	3
	Vapor	Weld	2	2
		No Weld	3	3
SAW	**Aqueous**	Weld	2	2
		No Weld	3	3
	Vapor	Weld	2	2
		No Weld	3	3
SCW	**Aqueous**	Weld	2	2
		No Weld	3	3
	Vapor	Weld	2	2
		No Weld	3	3

NOTE: 60 samples × 3 materials = 180 samples total.

Table 2.2.2.2. Task 2, FY09 Matrix 1: Prioritization of crevice samples for detailed analysis.

	# Samples at each condition		
Material	**SDW @ 90°C, 9.5 yr**	**SAW @ 90°C, 9.5 yr**	**SCW @ 90°C, 9.5 yr**
Alloy 625	3	3	3
Alloy C-4	3	3	3
Alloy G-3	3	3	3
Alloy 825	3	3	3

Table 2.2.2.3. Task 2, FY09 Matrix 2: Detailed analysis of priority 1 samples from Task 2, FY09, Matrix 1.

Environment[1]	T (°C)	# Samples at Each Condition and Exposure Time [2]			
		5 years	2.5 years	1 year	0.5 years
SDW	60	3	3	3	3
	90	3	3	3	3
SAW	60	3	3	3	3
	90	3	3	3	3
SCW	60	3	3	3	3
	90	3	3	3	3

[1] Only environments where crevice corrosion was observed in the activity described in Task 2, FY09, Matrix 1 will be examined.

[2] Samples will be examined starting with the 5 year exposures and working towards shorter exposure times. If no crevice attack is observed for two consecutive time periods, then the remaining samples at shorter time periods may not be inspected.

Table 2.2.2.4. Task 2, FY10, Matrix 1: Detailed Analysis of Priority 2,3 and 4 Samples from Task 2, FY09, Matrix 1

Environment[1]	T (°C)	# Samples at Each Condition and Exposure Time[2]			
		5 years	2.5 years	1 year	0.5 years
SDW	60	3	3	3	3
	90	3	3	3	3
SAW	60	3	3	3	3
	90	3	3	3	3
SCW	60	3	3	3	3
	90	3	3	3	3

[1] Only environments where crevice corrosion was observed in the activity described in Task 2, FY09, Matrix 1 will be examined.

[2] Samples will be examined starting with the 5 year exposures and working towards shorter exposure times. If no crevice attack is observed for two consecutive time periods, then the remaining samples at shorter time periods may not be inspected.

Table 2.2.2.5. Task 2, FY10, Matrix 2: Scoping experiments to characterize Alloy 22 crevice corrosion at open circuit potential.

	# Samples at Each Condition and Exposure Time			
Environment	**1 month**	**2 months**	**4 months**	**8 months**
0.1 *m* $CaCl_2$ @ 90°C	3	3	3	3
0.5 *m* $CaCl_2$ @ 90°C	3	3	3	3
1 *m* $CaCl_2$ @ 90°C	3	3	3	3
5 *m* $CaCl_2$ @ 90°C	3	3	3	3
6 *m* NaCl @ 90°C	3	3	3	3
6 *m* NaCl + 0.5 *m* KNO_3 @ 90°C	3	3	3	3

Table 2.2.2.6. Task 2, FY11-17, Matrix 1: Rough order estimate of long-term crevice coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29 (2,952 samples).

$[Cl^-]$ / *m*	$[NO_3^-]$ / *m*	Other composition	T (°C)	# Samples of each material analyzed at each time period								
				0.1 yr	**0.25 yr**	**0.5 yr**	**1 yr**	**2 yr**	**5 yr**	**10 yr**	**20 yr**	**Extra**
1	0	-	30	-	-	-	3	3	3	5	5	5
1	0	-	60	-	-	-	3	3	3	5	5	5
1	0	-	90	-	-	-	3	3	3	5	5	5
1	0	-	98	-	-	-	3	3	3	5	5	5
0.1	0	-	90	-	-	-	3	3	3	5	5	5
10	0	-	60	-	-	-	3	3	3	5	5	5
1	0.01	-	30	-	-	-	3	3	3	5	5	5
1	0.1	-	60	-	-	-	3	3	3	5	5	5
1	1	-	90	-	-	-	3	3	3	5	5	5
-	-	SAW	30	-	-	-	3	3	3	5	5	5
-	-	SAW	60	-	-	-	3	3	3	5	5	5
-	-	SAW	90	-	-	-	3	3	3	5	5	5
-	-	SDW	30	-	-	-	3	3	3	5	5	5
-	-	SDW	60	-	-	-	3	3	3	5	5	5
-	-	SDW	90	-	-	-	3	3	3	5	5	5
-	-	SCW	30	-	-	-	3	3	3	5	5	5
-	-	SCW	60	-	-	-	3	3	3	5	5	5
-	-	SCW	90	-	-	-	3	3	3	5	5	5
0	0	Distilled Water	90	-	-	-	3	3	3	5	5	5
1	0	1 ppm Pb	90	-	-	-	3	3	3	5	5	5
1	0	10 ppm Pb	90	-	-	-	3	3	3	5	5	5
1	0.1	10 ppm Pb	90	-	-	-	3	3	3	5	5	5
1	0	0.02 *m* F	90	-	-	-	3	3	3	5	5	5
1	0	0.2 *m* F	90	-	-	-	3	3	3	5	5	5
1	0.1	0.2 *m* F	90	-	-	-	3	3	3	5	5	5
1	0	0.02 *m* HCO_3^-	90	-	-	-	3	3	3	5	5	5
1	0	0.2 *m* HCO_3^-	90	-	-	-	3	3	3	5	5	5

[Cl^-] / *m*	[NO_3^-] / *m*	Other composition	T (°C)	# Samples of each material analyzed at each time period								
				0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
1	0.1	0.2 *m* HCO_3^-	90	-	-	-	3	3	3	5	5	5
1	0	4 mg/l SiO_3^{2-}	90	-	-	-	3	3	3	5	5	5
1	0	40 mg/l SiO_3^{2-}	90	-	-	-	3	3	3	5	5	5
1	0.1	40 mg/l SiO_3^{2-}	90	-	-	-	3	3	3	5	5	5
-	-	TBD-1	-	-	-	-	3	3	3	5	5	5
-	-	TBD-2	-	-	-	-	3	3	3	5	5	5
-	-	TBD-3	-	-	-	-	3	3	3	5	5	5
-	-	TBD-4	-	-	-	-	3	3	3	5	5	5
-	-	TBD-5	-	-	-	-	3	3	3	5	5	5
-	-	TBD-6	-	-	-	-	3	3	3	5	5	5
-	-	TBD-7	-	-	-	-	3	3	3	5	5	5
-	-	TBD-8	-	-	-	-	3	3	3	5	5	5
-	-	TBD-9	-	-	-	-	3	3	3	5	5	5
-	-	TBD-10	-	-	-	-	3	3	3	5	5	5

Table 2.2.2.7. Task 2, FY11-17, Matrix 2: Rough order estimate of long-term crevice coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (1700 samples, including TBDs).

Envrionments (10 total): 1 *m* NaCl, 90°C, neutral pH 1 *m* NaCl + 0.1 *m* $NaNO_3$, 90°C, neutral pH SAW, 90°C SCW, 90°C										
		# Samples analyzed at each time period, per environment, per material								
Material	Condition	0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
Alloy 22	Black Anneal	-	-	-	6	6	6	6	6	10
Alloy 22	LPB	-	-	-	3	3	3	3	3	5
Alloy 22	Simulated weld root	-	-	-	3	3	3	3	3	5
Alloy 22	Simulated HAZ	-	-	-	3	3	3	3	3	5
Alloy 22	Galvanic couple 316SS*	-	-	-	3	3	3	3	3	5
Alloy 22	Galvanic couple Titanium Gr. 7*	-	-	-	3	3	3	3	3	5
Alloy 22	Deliquescent pre-exposure	-	-	-	3	3	3	3	3	5
C276	Annealed	-	-	-	3	3	3	3	3	5
Ni	Annealed	-	-	-	3	3	3	3	3	5
Nichrome 80-20	Annealed	-	-	-	3	3	3	3	3	5
Ni-20 Cr-7 Mo	Annealed	-	-	-	3	3	3	3	3	5
Ni-15 Cr-7 Mo	Annealed	-	-	-	3	3	3	3	3	5
316 SS	Annealed	-	-	-	3	3	3	3	3	5
Future material	TBD	-	-	-	3	3	3	3	3	5
Titanium Gr7/28/29	Weld	-	-	-	3	3	3	3	3	5
Titanium Grade 28	Annealed	-	-	-	3	3	3	3	3	5
TBD-1	-	-	-	-	3	3	3	3	3	5
TBD-2	-	-	-	-	3	3	3	3	3	5
TBD-3	-	-	-	-	3	3	3	3	3	5
TBD-4	-	-	-	-	3	3	3	3	3	5
TBD-5	-	-	-	-	3	3	3	3	3	5

Table 2.2.2.8. Task 2, FY11-17, Matrix 3: Rough order estimate of crevice corrosion initiation and propagation testing under open circuit conditions for Alloy 22.

Goal	Notes	Approximate number of Samples	Approximate Duration
Crevice behavior in $CaCl_2$	-	120 (3 temperatures, 2 environments, 5 time steps[1], 4 samples per time step)	5 years
Crevice behavior in NaCl	-	80 (2 temperatures, 2 environments, 5 time steps[1], 4 samples per time step)	5 years
Effect of nitrate on rate and total extent of crevice attack	-	60 (1 temperature, 1 base environment, 3 nitrate levels, 5 time steps[1], 4 samples per time step)	5 years
Effect of crevice former material	materials include ceramic, metal and rock	60 (1 temperature, 1 environment, 3 crevice materials 5 time steps[1], 4 samples per time step)	5 years
Effect of force (or torque) on crevice former	-	60-100 (1 temperature, 1 environment, 3-5 levels of force, 5 time steps[1], 4 samples per time step)	5 years
Effect of thermal or mechanical processing	LPB and simulated weld root and weld HAZ, black anneal	320 (2 temperatures, 2 environments, 4 processing conditions, 5 time steps[1], 4 samples per time step)	5 years
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be required to improve confidence and reduce uncertainties.	100	5 years

[1] Samples removed at 0.25, 0.5, 1, 2 and 5 years.

2.2.3 Task 3: Critical Potential of Alloy 22

The objectives of this task are to improve confidence in crevice corrosion initiation models and predictions; improve understanding of the influence of physical crevice parameters (force, crevice material) and chemical environment on critical potential; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for localized corrosion behavior of Alloy 22 described in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Testing will be performed in FY08 to reproduce data that were excluded from the model for localized corrosion initiation of Alloy 22 (Table 2.2.3.1). These environments resulted in either very high values of E_{RCREV} or no observation of crevice corrosion following the test. Replication of the experiments will improve confidence that the data have been appropriately omitted from the model.

A test activity will be initiated in FY09 and continued through FY11 to determine the threshold values for temperature, chloride ion concentration and nitrate ion concentration where crevice corrosion initiates during a cyclic polarization (CP) experiment (Tables 2.2.3.2, 2.2.3.3, and part of 2.2.3.4). This information will allow the construction of a response surface that bounds the environmental conditions where crevice corrosion could occur in chloride-bearing electrolytes. In temperature, chloride concentration and nitrate concentration space, environments below the response surface would be excluded from the possibility of crevice corrosion and it would not be necessary to use a predictive model for those environments. After the response surface is defined, it will be possible to systematically map-out the repassivation potential response for values of temperature, chloride concentration and nitrate concentration where crevice corrosion can be initiated.

Two criteria can be used for determining threshold values of the environmental parameters. If a transition from no observed crevice corrosion to observed crevice corrosion occurs, then the threshold will have been identified. If a maximum value for repassivation potential is attained, then the threshold will have been identified. A candidate maximum value for repassivation potential is 600 mV vs saturated silver chloride electrode, as this value has been used by the Project in development of the LC model.

Testing will start at high temperature with 1 *m* chloride. If the sample does not exhibit crevice corrosion or the repassivation potential is above the threshold value, then the next highest chloride concentration will be tested. Otherwise, the next lowest chloride concentration will be tested. Once a threshold chloride concentration has been determined for a particular temperature the lowest chloride concentration that did not result in crevice corrosion or had a repassivation potential above the threshold value will be run two more times (a total of 3) to verify that the threshold has been identified. If the replicate runs result in a repassivation potential below the

threshold value and the sample exhibits crevice corrosion, then the sequence will continue with the next lowest chloride concentration.

Testing at the next lowest temperature in the matrix will begin with the minimum chloride concentration level that resulted in crevice corrosion and a repassivation potential below the threshold value at the previous temperature. In this fashion, the number of samples tested will be kept to a minimum while acquiring the desired data. Following this approach, it is reasonable to assume that approximately 5 to 7 tests will be performed for each temperature (4 tests if the threshold chloride concentration level is 1-off from that of the previous temperature. 6 tests if the threshold chloride concentration level is 3-off from the previous temperature). Some of the conditions will require additional tests (assume 3 conditions with 3 extra tests each) and the first temperature investigation may require on the order of 10 tests. Thus, the expected number of tests for each matrix will be ((4+6)/2) * 6 + 3*3 + 10 ≈ 49 tests. The TBD tests are to handle testing in concentrations higher than 5 *m* chloride. For the lower temperatures (where a high chloride concentration may be required to initiate corrosion) a test may be performed at the solubility limit of sodium chloride.

The threshold value study for temperature, chloride concentration and nitrate concentration will be continued in FY11-17 to include base environments of 0.01 *m* nitrate and 1 *m* nitrate (Table 2.2.3.4). Based on the results of these studies a systematic test program will be performed to characterize the relationship between localized corrosion parameters (particularly E_{RCREV}) and the environmental parameters of temperature, chloride concentration, and nitrate concentration (Table 2.2.3.5). The range of environments will only include conditions more aggressive than the threshold conditions determined via the testing summarized in Tables 2.2.3.3 and 2.2.3.4.

Additional studies performed in FY11-17 will examine other parameters that may influence the localized corrosion behavior of Alloy 22 including silicate, bicarbonate, other anions, surface finish, crevice material, crevice force (or torque), and cation species (Table 2.2.3.5). These testing activities will typically involve electrochemical testing such as CP or Tsujikawa-Hisamatsu electrochemical (THE) tests and each activity will require approximately six months to two years to complete, depending on the number parameters being examined.

Table 2.2.3.1. Task 3, FY08 Matrix 1: Electrochemical testing of Alloy 22 crevice corrosion behavior to reproduce critical potential data that were excluded from the localized corrosion model (General and Localized Corrosion of the Waste Package Outer Barrier (SNL 2007 [DIRS 178519])).

		Number of Samples by Experiment Type	
Environment	**T (°C)**	**THE[1]**	**CP**
6m NaCl + 0.3m KNO_3	80	3	3
0.0005 *m* NaCl	60	3	3
4 *m* KCl + 4 *m* NaCl + 0.4 *m* KNO_3 + 0.4 *m* $NaNO_3$	90	3	3
5 *m* $CaCl_2$	45	3	3
1.25 *m* NaCl	60	3	3
4 *m* NaCl	45	3	3
1 *m* NaCl + 0.15 *m* KNO_3	80	3	3
3.5 *m* NaCl + 0.525 *m* KNO_3	100	3	3
3.5 *m* NaCl + 0.525 *m* KNO_3	60	3	3
4 *m* KCl + 4 *m* NaCl + 2 *m* KNO_3 + 2 *m* $NaNO_3$ + 0.0001 *m* HCl	110	3	3
TBD[2]	-	3	3
TBD[2]	-	3	3
TBD[2]	-	3	3

[1] A maximum of three tests will be performed using the THE method. If sufficient information is gained from the CP experiments, the THE testing may be omitted or deferred or the number of tests reduced.

[2] The TBD tests are placeholders for testing that may be required to either (1) resolve any inconclusive results from the base matrix, or (2) improve the characterization of other environmental conditions in order to increase confidence in the LC model.

Table 2.2.3.2. Task 3, FY09, Matrix 1 – Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0.1 m NO_3^- with temperatures between 20°C and 95°C.

	# Samples Tested for Each Condition										
	Cl^- Concentration / *m*										
T / ºC	5	2	1	0.1	0.01	0.001	10^{-4}	10^{-5}	10^{-6}	TBD	TBD
~95	-	-	1	1	?	?	?	?	?	—	—
90	?	?	?	?	?	?	?	?	?	—	—
75	?	?	?	?	?	?	?	?	?	—	—
60	?	?	?	?	?	?	?	?	?	—	—
45	?	?	?	?	?	?	?	?	?	—	—
30	?	?	?	?	?	?	?	?	?	—	—
20	?	?	?	?	?	?	?	?	?	—	—

Table 2.2.3.3. Task 3, FY10, Matrix 1 – Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0 m NO_3^- with temperatures between 20°C and 95°C.

	# Samples Tested for Each Condition										
	Cl^- Concentration / *m*										
T / °C	5	2	1	0.1	0.01	0.001	10^{-4}	10^{-5}	10^{-6}	TBD	TBD
~95	-	-	1	1	?	?	?	?	?	—	—
90	?	?	?	?	?	?	?	?	?	—	—
75	?	?	?	?	?	?	?	?	?	—	—
60	?	?	?	?	?	?	?	?	?	—	—
45	?	?	?	?	?	?	?	?	?	—	—
30	?	?	?	?	?	?	?	?	?	—	—
20	?	?	?	?	?	?	?	?	?	—	—

Table 2.2.3.4. Task 3, FY11-17, Matrix 1: Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of Alloy 22.

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Determine threshold values for $[Cl^-]$ for 0.01 *m* NO_3^-.	Same approach as Task 3, FY09, Matrix 1	49 (See Task 3, FY09, Matrix 1 for sample # estimate)	1 year
Determine threshold values for $[Cl^-]$ for 1 m NO_3^-.	Same approach as Task 3, FY09, Matrix 1	49 (See Task 3, FY09, Matrix 1 for sample # estimate)	1 year
Systematic map of $[Cl^-]$, $[NO_3^-]$ and temperature dependencies of E_{RCREV}	Design of experiments approach will be used. Min values based on studies of threshold values	90 (20 <temperature< 95, 0 < $[Cl^-]$ < Sol. Limit, 0 < $[NO_3^-]$ < 1 m, 30 samples X 3 replicates)	2 years
Effect of silicates on E_{RCREV}	At least two base environments considered: Cl^- only and Cl^- + NO_3^-.	18 (2 base environments, 3 silicate concentrations, 3 replicates)	0.5 years
Effect of bicarbonate on E_{RCREV}	At least two base environments considered: Cl^- only and Cl^- + NO_3^-.	18 (2 base environments, 3 bicarbonate concentrations, 3 replicates)	0.5 years

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Effect of other anions (e.g., SO_4^{2-}, F^-) on E_{RCREV}	At least two base environments considered: Cl^- only and $Cl^- + NO_3^-$.	36 (2 base environments, 2 anion types, 3 anion concentrations, 3 replicates)	1 year
Effect of surface finish	Smooth vs. rough (e.g., 120 grit, 600 grit or mirror polish), black anneal	54 (2 environments, 2 temperatures 4 surface finishes, 3 replicates (6 for black anneal))	1 year
Effect of crevice former material	materials include ceramic, metal and rock	9 (1 base environment, 3 crevice materials, 3 replicates)	0.5 years
Effect of force (or torque) on crevice former	-	15 (1 environment, 5 force levels, 3 replicates)	0.5 years
Effect of cations on Ercrev	Systematic study of Ca (Mg) vs. Na (K) effects on E_{RCREV}, with or without NO_3^-	24 (2 temperatures, 2 cation types, 2 concentrations, with/w/out NO_3^-, 3 replicates)	1 year
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	100	2 years

[1] All tests in this matrix are either CP or THE experiments (or a derivation thereof).

2.2.4 Task 4: Open Circuit Potential of Alloy 22

This task will improve confidence in crevice corrosion initiation models and predictions; improve understanding of the influence of surface finish and oxide properties, physical environment (contact with tuff) and chemical environment on open circuit potential; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for localized corrosion behavior of Alloy 22 described in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

The Alloy 22 open circuit testing to be performed in FY08 (Table 2.2.4.1) will address: (1) electrolytes where previous instabilities have been observed, (2) behavior of open circuit potential in concentrated chloride, and (3) behavior of open circuit potential in dilute chloride. Previous open circuit measurements in a subset of the electrolytes tested showed significant instability. Some of these environments will be tested in this program to determine if the same instability is observed or if a cleaner signal can be obtained by running carefully isolated electrochemical measurements for each sample. The electrolytes to be tested are given in the first three rows of Table 2.2.4.1. The model for open circuit potential of Alloy 22 predicts a low dependence of open circuit potential on chloride concentration for concentrated electrolytes. Additional data from testing in concentrated electrolytes will improve confidence that the model appropriately represents the open circuit behavior. The three environments selected for this testing are given on rows 4-6 in Table 2.2.4.1. The model for open circuit potential of Alloy 22 predicts that low chloride concentrations will result in high open circuit values. Although this is a conservative treatment of open circuit potential, having additional data for low concentration electrolytes will aid in understanding the likelihood of localized corrosion under these conditions. The environments for this portion of the testing are listed in rows 7-10 in Table 2.2.4.1.

A systematic study will be undertaken to map out the dependence of open circuit potential and cathodic current density on pH, temperature, chloride concentration and nitrate concentration (Table 2.2.4.2). This will improve confidence in the current representation of open circuit potential in the LC model and will provide a framework for investigating secondary effects on Alloy 22 open circuit potential. Knowledge of the cathodic kinetics as a function of these parameters will lend insight into the ability of the Alloy 22 to sustain localized corrosion activity.

The possibility exists that the oxide structure or composition may be different following the thermal pulse and exposure to deliquescent conditions compared to oxides formed at room temperature. Such a change in the oxide is most likely to express itself as a change in the open circuit potential. The effects of exposure to deliquescent conditions on the open circuit behavior of Alloy 22 in seepage-relevant environments will be investigated by performing pre-exposures in a deliquescent environment followed by open circuit testing in an aqueous electrolyte (Table 2.2.4.3). If the open circuit potential of samples with varying exposure history is significantly different upon initial immersion in an electrolyte, this testing will also identify how the

difference in potentials evolves with time of exposure to the electrolyte. This task will be closely coupled with the oxide characterization task such that the impact of deliquescent conditions will be known both in terms of electrochemical behavior and oxide structure and oxide chemistry. Cathodic kinetics will also be measured to determine if pre-exposure to deliquescent conditions impacts the capacity of the oxide to support cathodic reactions.

Out year studies performed in FY11-17 will examine other parameters that may influence the open circuit potential of Alloy 22 including silicate, bicarbonate, other anions, surface finish, and cation species (Table 2.2.4.5). These testing activities will typically be intermediate to long term in duration (e.g., 2-5 years).

Table 2.2.4.1. Task 4, FY08 Matrix 1: Experiments to reproduce data from conditions where a large degree of variability was observed in the open circuit response.

Material Condition	Environment (Naturally Aerated Brines)	T (°C)	Approximate pH	# Samples	Cell # from bench top Ecorr tests
MA	5m $CaCl_2$ + 0.05m $Ca(NO_3)_2$	90	5	3	14
MA	5m $CaCl_2$ + 0.5m $Ca(NO_3)_2$	90	4.2	3	15, 21
MA	SCW	90	8.1	3	16
MA	7.5 m $CaCl_2$	90	Neutral	3	—
MA	10 m $CaCl_2$	90	Neutral	3	—
MA	10 m $CaCl_2$ + 10 m KNO_3	90	Neutral	3	—
MA	0.1 m NaCl	90	Neutral	3	—
MA	0.01 m NaCl	90	Neutral	3	—
MA	0.001 m NaCl	90	Neutral	3	—
MA	0.01 m NaCl + 0.01 m $NaNO_3$	90	Neutral	3	—
TBD	—	—	—	3	—
TBD	—	—	—	3	—

NOTE: One sample from each environment will have a dedicated potentiostat and E_{CORR} will be monitored continuously (or near continuously), or, based on equipment limitations, the potentiostat will be rotated among the three replicate samples with each sample monitored for a continuous fashion for 1/3 of the total test time. For example, the potentiostat could be rotated every week such that each sample has 1 week on and 2 weeks off in each 3 week cycle. Intermittent LPR or EIS experiments may be performed on some or all of the samples. Before the test is shut down a cathodic scan will be performed on at least a subset of the samples. The maximum current density should be limited as to avoid altering the oxide as post-test analysis will be a likely follow-on activity to this work.

Table 2.2.4.2. Task 4, FY09-11 Matrix 1: Systematic study of the effect of environmental variables on the open circuit potential of Alloy 22.

			# Samples at Each Condition					
Temperature (°C)	**$[Cl^-]$ / m**	**$[NO_3^-]$ / m**	**pH = 2**	**pH = 4**	**pH = 6**	**pH = 8**	**pH = 10**	**pH = TBD**
30	0.05	0	-	-	3	-	-	-
		0.1	-	-	3	-	-	-
	0.5	0	3	3	3	3	3	-
		1	-	-	3	-	-	-
	5	0	-	-	3	-	-	-
60	0.05	0	-	-	3	-	-	-
	0.5	0	-	3	3	-	-	-
		0.1	-	-	3	-	-	-
	5	0	-	-	3	-	-	-
		1	-	-	3	3	-	-
90	0.05	0	-	-	3	-	-	-
	0.5	0	3	3	3	3	3	-
		0.1	-	-	3	-	-	-
		1	3	3	3	3	3	-
	5	0	-	-	3	-	-	-
TBD			-	-	-	-	-	9

Table 2.2.4.3. Task 4, FY10 Matrix 1: Open circuit experiments to determine the effect of pre-exposure to deliquescent environments

			# Samples per Condition[1]	
Pre-exposure Conditions			Aqueous Environment	
Temperature (°C)	Contaminant	Gas Phase	SAW @ 90°C	SCW @ 90°C
150	none	Steam + Air	7	7
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	Steam + Air	7	7
205	none	Steam + Air	7	7
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	Steam + Air	7	7
Control samples / no pre-exposure			7	7

[1] One sample for each environment and temperature condition will have a dedicated potentiostat and open circuit potential will be monitored with intermittent EIS, LPR or other polarization. Three of the samples for each environment and temperature condition will be monitored periodically with E_{CORR}, LPR or EIS measurements. One of these three will be tested intermittently using a more severe galvanodynamic or potentiodynamic polarization to determine cathodic kinetics. The remaining three samples are witness coupons and will be used for oxide characterization studies. Samples will be removed at different time steps to be determined via examination of the trend in open circuit potential.

*** An extra sample will be prepared during the deliquescent exposure to be used as a control in the oxide studies and an extra sample that was not exposed to deliquescent conditions will also be provided to the oxide studies.**

Table 2.2.4.4. Task 4, FY11-17 Matrix 1: Rough order estimate of open circuit testing on Alloy 22 to determine the influence of secondary environmental parameters.

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Effect of contact with tuff	Ecorr will be measured in the presence of crushed tuff. Control samples come from Task 4, FY09-11, Matrix 1. Baseline environments will include: initially DI H_2O, a moderate Cl^- environment (e.g., pH 6, 0.5 m) and a $Cl^- + NO^-$ environment	21 (12, 9) (1 temperature, 3 environments, 4 echem samples per condition, 3 witness coupons per condition)	2 years
Effect of F^- on E_{CORR}	-	14 (8, 6) (1 temperature, 1 base environments, 2 F^- levels, 4 echem samples per condition, 3 witness coupons per condition)	2 years
Effect of HCO_3^- on E_{CORR}	-	14 (8, 6) (1 temperature, 2 HCO_3^- concentrations, 4 echem samples per condition, 3 witness coupons per condition)	2 years

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Effect of SiO_3^{2-} on E_{CORR}	-	14 (8, 6) (1 temperature, 2 SiO_3^{2-} concentrations, 4 echem samples per condition, 3 witness coupons per condition)	2 years
Effect of cations on E_{CORR}	Comparison of Na^+ to Ca^{2+}, one cation concentration, two base environments: Cl^- only and Cl^- + NO_3^-	28 (16, 12) (1 temperature, 2 base environments, 2 cation types, 4 echem samples per condition, 3 witness coupons per condition)	2 or 4 years (parallel or sequential testing)
Effect of surface finish on E_{CORR}	Surface finishes include black anneal, polished, thermal oxide and deliquescent exposure.	50 (20, 30)[2] (1 temperature, 1 environment, 4 surface finishes, 4 echem samples per condition (8 for black anneal), 6 witness coupons per condition (12 for black anneal).	5 years[2]
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	50	5 years

[1] The number of samples is given as *x (y, z)*; where *x* = the total number of samples, *y* = the number of electrochemically monitored samples (e.g., rods), and *z* = the number of witness samples (e.g., foils) to be removed at various time steps for archiving or surface analysis. Witness coupons may be removed dependent on analysis of the open circuit potential: that is, samples should be removed after the open circuit potential has apparently changed by a significant amount or after a prescribed length of time.

[2] It is anticipated that non-steady state surface finishes will evolve slowly toward a steady state structure and this process may require a significant exposure period. Thus, this activity has an increased number of witness samples and an increased duration compared to the other open circuit activities.

2.2.5 Task 5: Temperature Dependence of Alloy 22 and Titanium Alloys

The objectives of this task are to improve confidence in the representations of general corrosion rate temperature dependence in models and predictions; improve understanding of the influence of material condition, chemical environment and exposure time on temperature dependence; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for general corrosion behavior of Alloy 22 and titanium alloys described, respectively, in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]) and *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

The activities in this task will improve the understanding of the dependence of general corrosion rate on temperature for engineered barrier materials. Temperature dependence will be studied in two temperature regimes: (1) at or below boiling in environments representative of seepage conditions, (2) at or above boiling in environments that have a composition similar to deliquescent brines. A combination of electrochemical measurements and weight-loss measurements will be used to characterize the temperature dependence of the general corrosion rate. This task will provide for a direct comparison of electrochemical and weight-loss data and provide information on any relationship between the temperature dependence and exposure time. The electrolytes selected for this task include both SDW and SCW, thus providing for a measure of the impact of electrolyte composition on corrosion rate and temperature dependence.

In FY08 the focus of this task will be to make electrochemical measurements of Alloy 22 corrosion rate as a function of time and temperature (Table 2.2.5.1) and to start exposure tests on Alloy 22 for (Table 2.2.5.2) to determine temperature dependence via weight loss measurements.

In FY09 the Alloy 22 weight loss specimens from the seepage type environments will be weighed (Table 2.2.5.3) and the temperature dependence will be estimated and compared to results of the electrochemical testing. Exposure tests will be initiated on Titanium Grade 7, Titanium Grade 29 and Titanium Grade 28 in seepage type environments to measure temperature dependence (Table 2.2.5.4) and electrochemical testing will be performed on Titanium Grade 29 to measure corrosion rate as a function of time and temperature (Table 2.2.5.5). Additionally in FY09, testing will be initiated to determine the temperature dependence via weight loss of Alloy 22 and titanium materials in deliquescent type electrolytes (Table 2.2.5.6).

In FY10 the titanium weight loss specimens from the seepage type environments will be weighed (Table 2.2.5.7) and the temperature dependence will be estimated. Electrochemical testing will be performed on Titanium Grade 7 to measure corrosion rate as a function of time and temperature (Table 2.2.5.8).

Additionally in FY10, a test activity will be undertaken to measure the weight loss of Alloy 22 and titanium materials exposed to steam and humid environments (Table 2.2.5.9). Testing performed to date at LLNL and by the Center for Nuclear Waste Regulatory Analysis (CNWRA) has, in some cases, resulted in corrosion rates in the vapor space above an aqueous environment on the same order as the corrosion rates in the aqueous phase. These vapor phase test environments may have evolved to a more aggressive state than originally intended due to acid degassing and refluxing. This planned FY10 activity will use a carefully controlled experimental approach to determine the corrosion behavior in humid environments. These tests will also serve as controls for other experiments performed in out years where gas phase or solid contaminants may be introduced into the system.

The Alloy 22 and titanium specimens exposed to deliquescent type environments in FY09 will be weighed in FY10 (Table 2.2.5.10) and the temperature dependence estimated for deliquescent type environments. These data will provide bounding information on temperature dependence and corrosion rate and are intended to complement the deliquescent corrosion experiments performed under Task 8, *Alloy 22, Deliquescence Testing*.

In FY11-17, electrochemical testing will be performed on Titanium Grade 28 (or Titanium Grade 7 / Titanium Grade 28 / Titanium Grade 29 welds) to measure corrosion rate as a function of time and temperature (Table 2.2.5.11). This will complete the electrochemical testing for Alloy 22 and the titanium materials for assessing corrosion rate as a function of time and temperature.

Testing will be performed in the FY11-17 timeframe to determine the time and temperature dependence of Alloy 22 corrosion rate in a deliquescent brine type environment (Table 2.2.5.12). In addition to providing a measurement of temperature dependence as a function of time, this activity will give an indication of the propensity for general corrosion rate to be sustained or to decay under these conditions. Electrochemical measurements of Alloy 22 in deliquescent brine conditions will also be made (Table 2.2.5.13) to provide complimentary data to the weight loss study.

Table 2.2.5.1. Task 5, FY08 Matrix 1: Electrochemical testing to measure Alloy 22 temperature dependence in seepage type electrolytes.

Environment	# Samples at Each Condition[1]		
	30°C	60°C	90°C
SDW	6	6	6
SCW[2]	6	6	6

[1] One sample for each environment and temperature condition will have a dedicated potentiostat and open circuit potential will be monitored with intermittent EIS, LPR or other polarization. Four of the samples for each environment and temperature condition will be monitored periodically with E_{CORR}, LPR or EIS measurements. One sample for each environment and temperature condition will be tested intermittently using a more severe galvanodynamic or potentiodynamic polarization to determine cathodic kinetics.

[2] Although E_{CORR} measurements are also being made in SCW at 90°C in the task for *Alloy 22, Ecorr*, repeating some of the data in this task will provide a cross check on techniques and instrumentation. Additionally, having the instrumented samples in this matrix will provide a 1-to-1 comparison of electrochemical methods and weight-loss methods for determining temperature dependence.

Table 2.2.5.2. Task 5, FY08 Matrix 21: Exposure of Alloy 22 specimens to seepage type environments to determine temperature dependence.

Environment	# Samples at Each Condition[2]		
	30°C	60°C	90°C
SDW	10	10	10
SCW	10	10	10

[1] The weight loss exposure tests will be started in FY08. Based on the availability of material in the appropriate form (e.g., thick foil or thin sheet) this experiment may not start until late in FY08 and will continue into FY09. The samples will be analyzed for weight loss and change in physical appearance in FY09.

[2] Ten samples total will be exposed for each condition. Nine of the ten samples will be designated for weight-loss measurements and one of the ten samples will be designated for archiving and surface analysis.

Table 2.2.5.3. Task 5, FY09 Matrix 1: Weight loss measurements of Alloy 22 specimens exposed to seepage type electrolytes to determine temperature dependence.

Environment	# Samples at Each Condition[1]		
	30°C	60°C	90°C
SDW	10	10	10
SCW	10	10	10

[1] Ten samples total will be exposed for each condition. Nine of the ten samples will be designated for weight-loss measurements and one of the ten samples will be designated for archiving and surface analysis.

Table 2.2.5.4. Task 5, FY09 Matrix 2: Exposure of titanium materials to seepage type environments to determine temperature dependence.

Material	Environment	# Samples at Each Condition[1]		
		30°C	60°C	90°C
Titanium Grade 7	SDW	10	10	10
	SCW	10	10	10
Titanium Grade 29	SDW	10	10	10
	SCW	10	10	10
Titanium Grade 28	SDW	10	10	10
	SCW	10	10	10

[1] Ten samples total will be exposed for each condition. Nine of the ten samples will be designated for weight-loss measurements and one of the ten samples will be designated for archiving and surface analysis.

Table 2.2.5.5. Task 5, FY09 Matrix 3: Electrochemical testing to measure Titanium Grade 29 temperature dependence in seepage type electrolytes.

Environment	# Samples at Each Condition[1]		
	30°C	60°C	90°C
SDW	6	6	6
SCW	6	6	6

[1] One sample for each environment and temperature condition will have a dedicated potentiostat and open circuit potential will be monitored with intermittent EIS, LPR or other polarization. Four of the samples for each environment and temperature condition will be monitored periodically with E_{CORR}, LPR or EIS measurements. One sample for each environment and temperature condition will be tested intermittently using a more severe galvanodynamic or potentiodynamic polarization to determine cathodic kinetics.

Table 2.2.5.6. Task 5, FY09 Matrix 4: Short-term exposures (12 weeks) of Alloy 22 and titanium materials to deliquescent type electrolytes at 120°C, 150ºC and 180ºC to measure temperature dependence.

Material	Environment	# Samples at each Condition[1]		
		120°C	150°C	180°C
Alloy 22	$NaCl + NaNO_3 + KNO_3$	13	13	13
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	13	13	13
Titanium Grade 7	$NaCl + NaNO_3 + KNO_3$	6	6	6
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	6	6	6
Titanium Grade 29	$NaCl + NaNO_3 + KNO_3$	6	6	6
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	6	6	6

[1] One sample from each material-environment-temperature condition will be archived for surface analysis. The remainder of the samples will be designated for weight loss analysis.

Table 2.2.5.7. Task 5, FY10 Matrix 1: Weight loss measurements of titanium materials exposed to seepage type environments to determine temperature dependence.

Material	Environment	# Samples at Each Condition[1]		
		30°C	60°C	90°C
Titanium Grade 7	SDW	10	10	10
	SCW	10	10	10
Titanium Grade 29	SDW	10	10	10
	SCW	10	10	10
Titanium Grade 28	SDW	10	10	10
	SCW	10	10	10

[1] Ten samples total will be exposed for each condition. Nine of the ten samples will be designated for weight-loss measurements and one of the ten samples will be designated for archiving and surface analysis.

Table 2.2.5.8. Task 5, FY10 Matrix 2: Electrochemical testing to measure Titanium Grade 7 temperature dependence in seepage type electrolytes.

Environment	# Samples at Each Condition[1]		
	30°C	60°C	90°C
SDW	6	6	6
SCW	6	6	6

[1] One sample for each environment and temperature condition will have a dedicated potentiostat and open circuit potential will be monitored with intermittent EIS, LPR or other polarization. Four of the samples for each environment and temperature condition will be monitored periodically with EOC, LPR or EIS measurements. One sample for each environment and temperature condition will be tested intermittently using a more severe galvanodynamic or potentiodynamic polarization to determine cathodic kinetics.

Table 2.2.5.9. Task 5, FY10 Matrix 3: Weight loss testing of Alloy 22 and titanium materials exposed to steam and humid environments.

Material	Environment	# Samples at each condition	
		60°C	180°C
Alloy 22	Steam	-	3
	90% RH	3	-
Titanium Grade 7	Steam	-	3
	90% RH	3	-
Titanium Grade 29	Steam	-	3
	90% RH	3	-

Table 2.2.5.10. Task 5, FY10 Matrix 4: Weight loss measurements of Alloy 22 and titanium materials exposed to deliquescent type electrolytes at 120°C, 150ºC and 180ºC to measure temperature dependence.

Material	Environment	# Samples at each Condition[(1)]		
		120°C	150°C	180°C
Alloy 22	$NaCl + NaNO_3 + KNO_3$	13	13	13
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	13	13	13
Titanium Grade 7	$NaCl + NaNO_3 + KNO_3$	6	6	6
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	6	6	6
Titanium Grade 29	$NaCl + NaNO_3 + KNO_3$	6	6	6
	$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	6	6	6

[(1)] One sample from each material-environment-temperature condition will be archived for surface analysis. The remainder of the samples will be designated for weight loss analysis.

Table 2.2.5.11. Task 5, FY11-17 Matrix 1: Electrochemical testing to measure Titanium Grade 29 (or Grade 7 / Grade 28/Grade 29 welds) temperature dependence in seepage type electrolytes.

Environment	# Samples at Each Condition[1]		
	30°C	60°C	90°C
SDW	6	6	6
SCW	6	6	6

[1] One sample for each environment and temperature condition will have a dedicated potentiostat and open circuit potential will be monitored with intermittent EIS, LPR or other polarization. Four of the samples for each environment and temperature condition will be monitored periodically with EOC, LPR or EIS measurements. One sample for each environment and temperature condition will be tested intermittently using a more severe galvanodynamic or potentiodynamic polarization to determine cathodic kinetics.

Table 2.2.5.12. Task 5, FY11-17, Matrix 2: Medium duration testing of Alloy 22 in deliquescent brine chemistries

Alloy 22 immersed in $NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$				
Exposure time (years)	# Samples at each condition[(1)]			
	120°C	150°C	180°C	205°C
0.25	13	13	13	13
0.5	13	13	13	13
1	13	13	13	13

[(1)] One sample from each material-environment-temperature condition will be archived for surface analysis. The remainder of the samples will be designated for weight loss analysis.

Table 2.2.5.13. Task 5, FY11-17, Matrix 3: Electrochemical measurement of corrosion rate in deliquescent brine chemistries

Alloy 22 immersed in NaCl + $NaNO_3$ + KNO_3 + $Ca(NO_3)_2$			
# Samples at each condition[1]			
120°C	150°C	180°C	205°C
4	4	4	4

[1] Three of the four samples at each temperature will be monitored periodically using LPR and/or EIS measurements; for these experiments a 2 or 3 electrode technique may be used. The same three samples will also be periodically monitored versus a reference electrode to determine open circuit potential. The fourth sample will be used to determine cathodic kinetics using a higher magnitude polarization (e.g., 100's of mV instead of 10's of mV).

2.2.6 Task 6: Critical Potential of Titanium Alloys

The objectives of this task are to improve confidence in the understanding of titanium localized corrosion behavior in anticipated repository environments; improve understanding of the influence of material condition and chemical environment on critical potential; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for localized corrosion behavior of titanium alloys described in *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Localized corrosion of drip shield materials has been screened out based primarily on the exceptional localized corrosion resistance of titanium alloys in repository relevant environments. Testing will be performed to improve the fidelity in the understanding of how titanium corrosion behavior relates to chloride and fluoride concentrations in solution. This will be accomplished by performing a systematic study on the combined effects of chloride, fluoride and nitrate. The FY08 testing will focus on Titanium Grade 7 (Table 2.2.6.1) with FY09, FY10 and FY11 testing focusing on Titanium Grade 29, Titanium Grade 28 and welded titanium respectively (Tables 2.2.6.2 through 2.2.6.4).

Testing for secondary environmental effects such as silicate and bicarbonate effects will be performed in FY11-17 (Table 2.2.6.4). These experiments will improve understanding in the role of specific environmental parameters on localized corrosion behavior of titanium and will further improve confidence in the decision to screen out localized corrosion of the drip shield materials.

Table 2.2.6.1. Task 6, FY08 Matrix 1: Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 7.

CP/THE Testing of Titanium Grade 7 in $Cl/F/NO_3$ Environments					
$[Cl^-]$ (*m*)	$[F^-]$ (*m*)	$[NO_3^-]$ (*m*)	T (°C)	pH	# Tests[1]
0.1	0.01	-	90	Neutral	3
0.1	0.1	-	90	Neutral	3
0.1	1	-	90	Neutral	3
1	0.01	-	90	Neutral	3
1	0.1	-	90	Neutral	3
1	1	-	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.01	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.1	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	1	90	Neutral	3
TBD-1[3]					3
TBD-2[3]					3
TBD-3[3]					3

[1] The first two tests will be CP. If high variability is observed, a third CP test will be performed. If crevice corrosion is observed, the third test will be a THE.

[2] After the matrix of $[Cl^-]$ and $[F^-]$ is run, a combination of $[Cl^-]$ and $[F^-]$ will be chosen to investigate the inhibiting effects of NO_3^-. The concentrations will be chosen based on consideration of reproducibility and magnitude of the corrosion response.

[3] Additional experiments will be run if either of the following two conditions is met: (1) the initial matrix of experiments suggests that the range of concentrations should be expanded to fully characterize the system response, or (2) the system is not well behaved at one of the tested conditions and additional experiments are needed to reduce uncertainty.

Table 2.2.6.2. Task 6, FY09 Matrix 1: Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 29.

CP/THE Testing of Titanium Grade 29 in $Cl^-/F^-/NO_3^-$ Environments					
$[Cl^-]$ (*m*)	$[F^-]$ (*m*)	$[NO_3^-]$ (*m*)	T (°C)	pH	# Tests[1]
0.1	0.01	-	90	Neutral	3
0.1	0.1	-	90	Neutral	3
0.1	1	-	90	Neutral	3
1	0.01	-	90	Neutral	3
1	0.1	-	90	Neutral	3
1	1	-	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.01	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.1	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	1	90	Neutral	3
TBD-1[3]					3
TBD-2[3]					3
TBD-3[3]					3

[1] The first two tests will be CP. If high variability is observed, a third CP test will be performed. If crevice corrosion is observed, the third test will be a THE.

[2] After the matrix of $[Cl^-]$ and $[F^-]$ is run, a combination of $[Cl^-]$ and $[F^-]$ will be chosen to investigate the inhibiting effects of NO_3^-. The concentrations will be chosen based on consideration of reproducibility and magnitude of the corrosion response.

[3] Additional experiments will be run if either of the following two conditions is met: (1) the initial matrix of experiments suggests that the range of concentrations should be expanded to fully characterize the system response, or (2) the system is not well behaved at one of the tested conditions and additional experiments are needed to reduce uncertainty.

Table 2.2.6.3. Task 6, FY10 Matrix 1: Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 28.

CP/THE Testing of Titanium Grade 28 in $Cl^-/F^-/NO_3^-$ Environments					
$[Cl^-]$ (*m*)	$[F^-]$ (*m*)	$[NO_3^-]$ (*m*)	T (°C)	pH	# Tests[1]
0.1	0.01	-	90	Neutral	3
0.1	0.1	-	90	Neutral	3
0.1	1	-	90	Neutral	3
1	0.01	-	90	Neutral	3
1	0.1	-	90	Neutral	3
1	1	-	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.01	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	0.1	90	Neutral	3
0.1 or 1[2]	0.01, 0.1 or 1[2]	1	90	Neutral	3
TBD-1[3]					3
TBD-2[3]					3
TBD-3[3]					3

[1] The first two tests will be CP. If high variability is observed, a third CP test will be performed. If crevice corrosion is observed, the third test will be a THE.

[2] After the matrix of $[Cl^-]$ and $[F^-]$ is run, a combination of [Cl] and [F] will be chosen to investigate the inhibiting effects of NO_3^-. The concentrations will be chosen based on consideration of reproducibility and magnitude of the corrosion response.

[3] Additional experiments will be run if either of the following two conditions is met: (1) the initial matrix of experiments suggests that the range of concentrations should be expanded to fully characterize the system response, or (2) the system is not well behaved at one of the tested conditions and additional experiments are needed to reduce uncertainty.

Table 2.2.6.4. Task 6, FY11-17 Matrix 1: Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of titanium alloys and welds.

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Testing of Welds	Titanium 7/28/29 welds will be tested in Cl^-, $Cl^- + NO_3^{2-}$, and $Cl^- + F^- + NO_3^{2-}$ environments.	36 (See Task 6, FY09, Matrix 1 for details)	1 year
Effect of silicates on E_{RCREV} of Titanium Grade 7, Titanium Grade 29, Titanium Grade 28	-	18 (1 base environment, 2 silicate concentrations, 3 materials, 3 samples per condition)	0.5 years
Effect of bicarbonate on E_{RCREV} of Titanium Grade 7, Titanium Grade 29, Titanium Grade 28	-	18 (1 base environment, 2 bicarbonate concentrations, 3 materials, 3 samples per condition)	0.5 years
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	36	1 year

[1] All tests in this matrix are either CP or THE experiments (or a derivation thereof)

2.2.7 Task 7: Characterization of Alloy 22 and Titanium Oxides

The objectives of this task are to improve confidence in models and predictions that rely on long-term stability or repassivation characteristics of Alloy 22 and titanium oxides; improve understanding of the influence of material condition, exposure history, electrochemical potential, and chemical environment on oxide properties, structure and composition; and provide supplemental data, where appropriate, to performance margin analyses.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for general and localized corrosion behavior of Alloy 22 and titanium alloys described, respectively, in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]) and *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

The stability of the Alloy 22 waste package outer barrier has been effectively demonstrated through electrochemical and weight-loss measurements. However, confidence in the long-term stability of the protective oxide layer can be improved by examining the structure in the oxide and at the oxide-metal interface at nanometer dimensions. Several analysis techniques will be exercised to determine an optimized approach to characterizing specimens that have a large surface roughness. Samples from the LTCTF will be selected for examination to determine the structure and/or composition of the oxide films following long-term exposure to repository-relevant electrolytes (Table 2.2.7.1). This study will also provide baseline information and guidance to out year activities that examine specific processes that may define oxide stability. The developed approach to oxide characterization will then be applied to analysis of oxides from the 9.5 year exposure samples (Table 2.2.7.2).

The effect of exposure to deliquescent conditions on oxide characteristics will be studied by analyzing samples exposed at high temperature and high dew point for various durations (Table 2.2.7.3). These data will be used in conjunction with open circuit measurements on similarly treated specimens (Task 4) to develop understanding of how changes in the oxide properties impact open circuit behavior and, by inference, localized corrosion behavior.

The longer term (FY11-FY17) oxide characterization activities receive input, in the form of exposed samples, from the short, medium and long-term duration sample exposures in all other tasks. Specific activities are listed in Table 2.2.7.4 for this task; however, the prioritization of samples will be strongly influenced by the results of other measurements (e.g., weight loss, E_{CORR}, E_{RCREV}, optical or SEM observation, etc.). Detail for the analysis of samples exposed to aqueous environments following a deliquescent pre-treatment is given in Table 2.2.7.5. This activity will yield information on the propensity for the oxide to retain characteristics established during the high-temperature / high dewpoint period. Information will also be developed on the rate at which the oxide approaches an equilibrium structure and composition.

In addition to characterizing samples from other tasks, the oxide characterization task will include a systematic study of the electrochemical and chemical stresses that cause changes to the

structure and composition of the Alloy 22 oxide film (See Table 2.2.7.4). This latter task will provide fundamental understanding of the parameters that define stability for the oxide and will be a framework for understanding the results of other oxide characterization efforts.

Table 2.2.7.1. Task 7, FY08 Matrix 1: Characterization of Alloy 22 oxides on specimens exposed for 5 years in the LTCTF.

Number of Individual Sites, Tests, or Measurements to be Conducted				
	Exposure Conditions			
Tests / Measurements[1]	Control-1 SAW, 60°C, Vapor	Control-2 TBD[2]	SDW, 90°C, Aqueous	SCW, 90°C, Aqueous
SEM site selection	11	11	11	11
SEM EDS	11	11	11	11
SEM EBSD[3]	5	5	5	5
Topographic Mapping[4]	8	8	8	8
TEM cross-section	3	3	3	3
TEM EDS or EELS	3	3	3	3
TOF SIMS profile[3,4]	5	5	5	5
XPS profile[4]	3	3	3	3
Topographic Mapping[4]	8	8	8	8
TBD	5	5	5	5
TBD	5	5	5	5
TBD	5	5	5	5

[1] Characterization techniques and the general sequence of analysis, as shown in column 1:

- Surface imaging using variable incident energy Secondary Electron Microscopy (SEM) in combination with Electron Backscattered Diffraction (EBSD) and Energy Dispersive X-ray Fluorescence Spectroscopy (EDS). Electron microscopy is used to map candidate regions for subsequent cross-section sample extraction or depth profile analysis. Fiducial marking will be conducted using electron or ion stimulated deposition of carbon or platinum features. EBSD will provide local grain orientation for the alloy bulk. EDS will provide elemental composition of mapping within the energy-dependent excitation volume of incident beam. Focused ion beam (FIB) milling will be used for cross-section sample preparation.
- Mapping of surface topology using optical interferometry and atomic force microscopy (AFM). Surface topology will be used to further determine best suited regions for subsequent depth profile measurements by investigating SEM identified areas of the samples. The initial topological profile will be used in a multivariate analysis routine to adjust the depth index for the depth profile process. Post profiling topology measurements will also be made in order to create a final depth index.
- Structural and compositional analysis of the passive oxide using Transmission Electron Microscopy (TEM) of cross-sectioned samples with appropriate spectroscopic characterization (X-Fluorescence or Electron Energy Loss Spectroscopy) applied combined with multivariate statistical analysis.
- Time-of-flight Secondary Ion Mass Spectrometry (TOF-SIMS) combined with sputter depth profiling and multivariate statistical analysis. Candidate sites will be pre-screened with SEM and surface topology measurements based on a fiducial marking technique. Topographic maps will be used to index the absolute depth axis position for each pixel to facilitate a 3-dimensional compositional depth profile.
- X-ray Photoelectron Spectroscopy (XPS) combined with sputter depth profiling to gain insight into chemical speciation within the passive oxide.
- Post-depth profiling mapping of surface topology using optical interferometry and atomic force microscopy (AFM).

[2] A control sample may be used that has reduced surface roughness compared to the samples from the LTCTF. Data from this sample will aid in determining what information can be gained by the experimental protocol when unconstrained by interference from surface topography. This sequence of experiments will also help guide selection of material conditions for other corrosion testing (e.g., deliquescent, next-generation long-term exposures).

[3] Measurements support TOF-SIMS.

[4] Measurements support depth profiling analysis.

Table 2.2.7.2. Task 7, FY09 Matrix 1: Characterization of Alloy 22 oxides on specimens exposed for 9.5 years in the LTCTF.

Number of Individual Sites, Tests, or Measurements to be Conducted						
Tests / Measurements[1,2]	**Exposure Conditions[3]**					
	SAW, 9.5 years		**SDW, 9.5 years**		**SCW, 9.5 years**	
	60°C	**90°C**	**60°C**	**90°C**	**60°C**	**90°C**
SEM site selection	11	11	11	11	11	11
SEM EDS	11	11	11	11	11	11
SEM EBSD	5	5	5	5	5	5
Topographic Mapping	8	8	8	8	8	8
TEM cross-section	3	3	3	3	3	3
TEM EDS or EELS	3	3	3	3	3	3
TOF SIMS profile	5	5	5	5	5	5
XPS profile	3	3	3	3	3	3
Topographic Mapping	8	8	8	8	8	8
TBD	5	5	5	5	5	5
TBD	5	5	5	5	5	5
TBD	5	5	5	5	5	5

[1] The details for the analysis methods are provided in the FY08 test matrix (above).

[2] It should be noted that the same analyses are prescribed here as appear in the FY08 oxide characterization effort (*Task 7, FY08, Matrix 1*). The FY08 task is exploratory in nature and it is likely that the specific characterization activities used in the FY09 program will differ from those to some extent. For planning purposes it is reasonable to assume that the analyses used in FY09 will be of similar scope and require similar resources as to the matrix included here.

[3] The exposure conditions in this table differ from what was indicated in the interim deliverable for FY08 testing. In that testing document it was stated that the matrix of 5 year samples from SAW, SDW and SCW at 60ºC and 90ºC would be completed in FY09. However, analysis of the 9.5 years samples will yield a more definitive data set and will allow comparison of 5 year and 9.5 year oxides for SAW and SCW. Based on the outcome of these analyses, it may not be necessary to analyze the remainder of the 5 year samples.

Table 2.2.7.3. Task 7, FY10 Matrix 1: Characterization of Alloy 22 on specimens exposed to deliquescent conditions.

		# Samples at each condition					
		150°C			**205°C**		
		duration / weeks			**duration / weeks**		
solid contaminant	**atmosphere**	2	4	12	2	4	12
no contaminant	**steam + air**	1	1	1	1	1	1
$NaCl + NaNO_3 + KNO_3$	**steam + air**	1	1	1	1	1	1
$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$	**steam + air**	1	1	1	1	1	1

Table 2.2.7.4. Task 7, FY11-17 Matrix 1: Summary of oxide characterization activities in FY11-17.

Goal	Notes	Approximate number of Samples	Approximate Duration
Analyze samples from other FY11-17 tasks	Analysis procedures include SEM, FIB, TEM, AFM and surface analytical techniques (e.g., SIMS, XPS, AES, etc.)	20-30 / year (This is an estimate of a reasonable effort level for a PI to oversee in this area)	continuous starting in FY11
Analyze Alloy 22 oxides from samples exposed to aqueous solutions following deliquescent pre-exposure	See matrix below: *Task 7, FY11-17, Matrix 2*	32 (see notes)	1.5 years
Oxide stability studies	Characterize the effect of chemical and electrochemical stresses on the stability, composition and structure of Alloy 22 oxide and the metal/oxide interface. This activity will included electrochemical studies, surface analysis and possibly development of UHV or other techniques for oxide modification/manipulation.	10's to 100's of sites per year for modification and characterization	5 years

Table 2.2.7.5. Task 7, FY11-17 Matrix 2: Analysis of Alloy 22 oxides from samples exposed to aqueous solutions following deliquescent pre-exposure.

	# Samples per condition[1]							
	Aqueous Exposure Environment							
	SAW @ 90°C				SCW @ 90°C			
	Exposure period				Exposure period			
Pre-exposure condition	a[2]	b[3]	c[3]	d[3]	a[2]	b[3]	c[3]	d[3]
None (control)	1	1	1	1	1	1	1	1
150°C, NaCl + $NaNO_3$ + KNO_3 + $Ca(NO_3)_2$, steam+air	1	1	1	1	1	1	1	1
205ºC, no solid contaminant, steam+air	1	1	1	1	1	1	1	1
205ºC, NaCl + $NaNO_3$ + KNO_3 + $Ca(NO_3)_2$, steam+air	1	1	1	1	1	1	1	1

[1] Each condition has one associated sample. The sample may be analyzed with multiple techniques. Selection of specific techniques will be based on the results of the oxide characterization studies performed in FY08-FY10. A subset of the samples from the open circuit experiments described in *Task 4, FY10, Matrix 1* will be examined in FY11 (as described in the matrix). The remainder of the samples from *Task 4, FY10, Matrix 1* will be characterized in out-years, if necessary.

[2] Exposure period *a* is the unexposed control sample and therefore has a duration of zero.

[3] Exposure periods *b, c,* and *d* will be determined during the FY09 testing described in *Task 4, FY09, Matrix 1*. It may not be necessary to examine samples from all exposure periods; the decision will be made after consideration of the associated open circuit data and after initial analysis of select oxides (e.g., examination of the shortest and longest exposure periods).

2.2.8 Task 8: Alloy 22, Deliquescence Testing

The objectives of this task are to improve confidence in general and localized corrosion predictions for deliquescent conditions; improve understanding of the influence of temperature, chemical environment and exposure time on oxide stability and damage accumulation; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for general and localized corrosion behavior of Alloy 22 described in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]) and *Analysis Of Dust Deliquescence For FEP Screening* (SNL 2007 [DIRS 181267]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

Testing under carefully controlled deliquescent conditions requires development of the proper facilities and techniques. The approach to developing the methodologies for characterizing initiation and stifling under deliquescent conditions is given in Table 2.2.8.1. The results of these tests will provide guidance to the remaining activities under this task for assessing corrosion behavior under deliquescent conditions.

The relationship between initial salt loading and total accumulated damage (assessed by weight loss, resistance change or imaging techniques) will be determined for samples exposed to deliquescent conditions (Table 2.2.8.2). Specimens of Alloy 22 and less corrosion resistant materials (e.g., Nichrome) will be tested with various salt loadings to determine how much damage occurs for a given starting condition. Additional, confirmatory, tests may also be run in parallel or in series with these experiments where salt-loaded and creviced materials will be exposed in the environmental chamber then examined ex-situ for localized corrosion initiation. For each temperature investigated, an additional objective will be to determine the minimum nitrate to chloride ratio required to inhibit crevice corrosion. Creviced Alloy 22 and surrogate materials will be exposed to deliquescent conditions (at temperatures up to 205ºC) at the minimum nitrate to chloride ratio for which deliquescence is predicted to occur; this will be the least inhibitive environment that can form at that temperature. If localized corrosion is observed for this minimum nitrate to chloride ratio, then additional testing will be performed at higher nitrate-to-chloride ratios until a critical value is determined for inhibiting corrosion at a given temperature.

The ability of nitrate to inhibit localized corrosion in a chloride-bearing environment depends on establishing a sufficient concentration of nitrate (or nitrate to chloride ratio) within the occluded volume (either a pit or a crevice). A series of crevice corrosion experiments will be performed under aqueous conditions in which crevice corrosion will be initiated in a chloride solution then a series of nitrate inoculations will be made to the bulk solution. Either be potentiostatic holds or cyclic polarization scans (or THE tests) will be used to interrogate the behavior. For a potentiostatic hold experiment the transition from active to passive behavior will be used to assess the effect of the nitrate addition whereas for a cyclic polarization test the repassivation

potential will be used. The experiment described here is designed to add confidence that nitrate levels in a crevice can be established by the migration of nitrate. This activity is planned for FY09.

It is likely that the deliquescent environment will evolve with time to an even less aggressive composition than the starting salt assemblage. Testing will be performed to determine the evolution of brine chemistry for salts and salt assemblages exposed to high temperature, high dewpoint environments. The matrix for these tests is shown in Table 2.2.8.3.

The general corrosion rate of Alloy 22 under deliquescent conditions will be measured both by testing under inundated conditions in deliquescent brines (See Task 5, Tables 2.2.5.12 and 2.2.5.13) and by direct measurement of weight change due to exposure to deliquescent conditions (Table 2.2.8.4). Testing under inundated conditions will provide upper bounds on the sustainable general corrosion rate while testing under deliquescent conditions will generate a less conservative estimate of general corrosion behavior under these conditions. The two approaches together will provide an indication of the relative magnitude of corrosion (and accumulated damage) that can be supported in the presence and absence of a bulk electrolyte.

The effect of exposure to deliquescent conditions on oxide characteristics will be studied by analyzing samples exposed at high temperature and high dew point for various durations (Table 2.2.8.5). Samples from these exposures will be provided to the oxide characterization task (Task 7) and to the Alloy 22 open circuit behavior task (Task 4). The output from these activities will be an understanding of the influence of the deliquescent environment on open circuit behavior in seepage-type electrolytes and an understanding of how oxide characteristics depend on exposure history.

The testing in FY11-17 for this task will focus on long-term measurements and observations of material corrosion behavior under deliquescent conditions (Table 2.2.8.6). Alloy 22 and less corrosion resistant materials will be exposed for up to five years to deliquescent environments and localized corrosion initiation and propagation evaluated using both in-situ measurements and ex-situ post-test inspections. Weight-loss coupons will also be used in the testing to determine the average general corrosion rate under deliquescent conditions for extended exposure periods (e.g., five years).

Table 2.2.8.1. Task 8, FY08 Matrix 1: Evaluation of sample configurations and monitoring techniques for corrosion studies under deliquescent conditions.

Trial #	Experimental Details
1	Ribbon coupon with DCPD monitoring[1,3]
2	Creviced sample with DCPD monitoring[1,3]
3	Two-electrode assembly with applied potential[2,3]
4	Two-electrode assembly with crevice former and applied potential[2,3]
5	Two-electrode assembly with applied potential and DCPD[2,3]
6	Two-electrode assembly with crevice former, applied potential and DCPD[2,3]

[1] Multiple samples can be instrumented and monitored simultaneously such that a given experiment may include multiple materials or multiple salt loadings.

[2] For development purposes, it will be practical to only run one experiment at a time; however, these tests are driven and the length of the experiment will be determined by the equilibration period. The analogue in aqueous testing is the CP test where a 24-hr open circuit period defines the length of the experiment. In the case of high temperature testing, the most conservative experiment will likely be to impose a potential as soon as deliquescence has occurred.

[3] These experiments may be performed *non-Q* as they are only scoping in nature and are providing guidance for performing the Q test matrix below.

Table 2.2.8.2. Task 8, FY08 Matrix 2: Corrosion behavior under deliquescent conditions of Alloy 22 and less-corrosion resistant materials as a function of mass-loading, temperature and dewpoint.

Material	Loading[1] (µg-cm^{-2})	Contaminant	T[2] (°C)	Nominal Dewpoint[3] (°C)	# Samples[4]
Nichrome	10 to 500	4 salt assemblage	180 to 205	92, 95	5 replicates (20 samples)
Nichrome	TBD[5]	Collected dust	180 to 205	92, 95	5 replicates (5 samples)
Alloy 22	10 to 500	4 salt assemblage	180 to 205	92, 95	5 replicates (20 samples)

[1] The mass-loading of contaminant will initially be in the range 10 to 500 µg-cm^{-2}. The goal will be to differentiate the total accumulated damage for 2 or more mass loading values. Higher or lower values may be used if necessary to produce differentiable behavior. If test-to-test variability overwhelms the difference in response to varying contaminant levels, then the distribution of total accumulated damage will be reported for a nominal range of contaminant values rather than developing a relationship between contaminant loading and total damage.

[2] The initial temperature chosen for this experiment will be at the upper end of the temperature range given in the table unless experimental results from the scoping experiments provide compelling evidence that a different initial value should be used. If testing is inconclusive or the effects of temperature are deemed critical to the corrosion behavior then other experiments may be run at other temperatures in this range.

[3] The dewpoints chosen represent a pure steam atmosphere (T_D = 95°C) or a mixed steam plus dry gas (e.g., CO_2, O_2) atmosphere (T_D = 92°C).

[4] The number of samples is nominally for five replicates run at two different dew points and two mass loadings.

[5] A nichrome sample with collected dust will be run in order to determine if natural dust can initiate corrosion on a less corrosion resistant analogue to Alloy 22. The dust loading will be determined after knowledge is gained concerning system behavior by running experiments using salt assemblages.

Table 2.2.8.3. Task 8, FY08 Matrix 3: Evaluation of the stability of salt and salt assemblages in high-temperature, high dewpoint environments.

Effect of Temperature on Brine Chemistry at T_D = 92°C						
	# of Samples Held at Each Temperature[1]					
	150°C		180°C		205°C	
Composition	1 day	5 days	1 day	5 days	1 day	5 days
$CaCl_2$	2	2	2	2	2	2
$NaNO_3$	2	2	2	2	2	2
3 salt assemblage	2	2	2	2	2	2
4 salt assemblage	2	2	2	2	2	2
TBD[2]	2	2	2	2	2	2
TBD[3]	2	2	2	2	2	2

[1] Each sample will be measured for weight change and will be analyzed using XRD. If the required information is obtained from the 1-day experiments, the 5-day experiments may be omitted.
[2] If needed, a set of tests will be reproduced from the matrix to address variability in results.
[3] If needed, another salt or salt assemblage will be tested to strengthen understanding of brine evolution.

Table 2.2.8.4. Task 8, FY09 Matrix 1: Effect of Deliquescent conditions on oxide stability (provides comparison to weight loss studies from Task 5 and supplies samples to oxide characterization studies in Task 7).

		# Samples at each condition[1]					
		150°C			205°C		
		duration / weeks			duration / weeks		
solid contaminant	atmosphere	2	4	12	2	4	12
no contaminant	steam + air	6	6	6	6	6	6
NaCl + $NaNO_3$ + KNO_3	steam + air	6	6	6	6	6	6
NaCl + $NaNO_3$ + KNO_3 + $Ca(NO_3)_2$	steam + air	6	6	6	6	6	6

[1] 5 samples will be used for weight loss studies and one sample will be archived or used for surface analysis.

Table 2.2.8.5. Task 8, FY10 Matrix 1: Effect of deliquescent conditions on open circuit behavior in seepage environments (provides input to Tasks 4 and 7).

		# Samples at each condition[1]	
		Temperature	
Contaminant	Gas Phase	150°C	205°C
None	Steam + Air	15	15
NaCl + $NaNO_3$ + KNO_3 + $Ca(NO_3)_2$	Steam + Air	15	15

[1] Duration of exposure will be determined based on analysis of samples from Task 8, FY09, Matrix 1.

Table 2.2.8.6. Task 8, FY11-17 Matrix 1: Summary of long-term testing under deliquescent environments.

Goal	Notes	Approximate number of Samples	Approximate Duration
Determine crevice initiation/stifling behavior for Alloy 22, analogues and surrogates under deliquescent conditions using ex-situ inspection technique to characterize damage.	Examine samples for various exposure times to characterize induction time, estimate of damage accumulation rate, total accumulated damage and tendency for stifling & arrest.	120 samples (4 materials, 2 temperatures, 1 environment, 3 replicates, 5 time steps)	5 years[1]
Determine long-term general corrosion rate of Alloy 22, analogues and surrogates in deliquescent environments	Perform weight loss and characterize oxide after medium to long-term exposures.	144 samples (4 materials, 2 temperatures, 1 environment, 4 replicates[2], 5 time steps)	5 years[1]
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	100	5 years

[1] Current facilities may require multiple sequential experiments for each activity. Running more samples in parallel will either require new facilities or increasing the capacity of the current system.

[2] Weight-loss measurements will be performed on 3 samples; the fourth sample will be archived for surface analysis.

2.2.9 Task 9: Microbial Influenced Corrosion on Alloy 22

The objectives of this task are to improve confidence in the modeled and predicted effects of microbial activity on general and localized corrosion; improve understanding of the influence of temperature, humidity, physical and chemical environment and exposure time on microbial effects and oxide stability and damage accumulation; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the treatments of microbial influenced corrosion described in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS 178519]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Alloy 22 specimens tested in growth media for up to 57 months were previously examined using SEM and were found to have widespread attack consisting of pores with diameters typically in the 10 to 100 nm range. Samples were tested for various exposure times and the opportunity exists to examine the damage state in more detail to establish qualitative and perhaps quantitative relationships between exposure time and damage condition. This activity will examine the morphology of attack from two exposure periods to determine: (1) if the damage state can be quantitatively assessed using cross-sectioning techniques, (2) if the damage state can be differentiated for different exposure times and (3) the aspect ratio of the pores. These data could improve confidence that the damage state does not progress after a given exposure period even in nutrient rich media. Additionally, this task will provide information on the depth of attack caused by MIC under these exposure conditions; a bounding case for MIC. Table 2.2.9.1 provides the details of the specimens to be examined in FY08. This task will also serve to guide out-year activities for analysis of MIC specimens.

In FY09 a literature review will be performed and a testing apparatus designed to support MIC testing. In FY10 the MIC testing rig will be procured or developed and non-Q tests performed to support parameter development and equipment qualification for Q work to being in FY11.

Although the specifics of the long-term MIC testing will not be developed until FY09/10, the general scheme will be to expose samples to repository relevant electrolytes (in a high-humidity environment) in the presence of solid contaminants (dust, rock, corrosion products) and inoculate the system with representative and relevant microbe consortia. To increase the aggressiveness of the test it may be necessary to re-inoculate the system on a periodic basis, thus ensuring ample opportunity for microbe growth. Samples to evaluate general corrosion and localized corrosion processes will be included in the test matrix. Some of the samples will be witness coupons used as inputs into the oxide characterization task. A sampling scheme will be employed to gauge the ability of microbes to thrive under the test conditions. A general outline of the extent of testing to be performed in FY11-17 is given below in Table 2.2.9.2.

Table 2.2.9.1. Task 9, FY08 Matrix 1: Analysis of MIC samples from testing in growth media.

Exposure Time / Months	# of Analyses Performed Per Technique	
	Imaging / automated analysis[1, 2]	FIB + SEM[2]
17	1	5
57	1	5

[1] The effort required for imaging and analysis is not easily expressed in terms of a single value. Imaging work will be performed to gain an understanding of the density and dimensions of micro-pits on a representative sample for each listed exposure time.

[2] The analysis may be performed *non-Q* as this is a scoping activity intended to inform out-year test programs.

Table 2.2.9.2. Task 9, FY11-17 Matrix 1: Rough order estimate of out year MIC testing to be performed on Alloy 22.

Temperature (°C)	Humidity (%RH)	Contaminants	Samples[1]
25	60	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	90	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	100	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
50	60	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	80	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	100	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
90	60	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	80	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
	100	rock	9, 3, 3, 1
		rock + salts	9, 3, 3, 1
3 TBD environments with 100 additional samples			

[1] Samples are given as: *a, b, c, d* where: a = # samples for general corrosion studies, b = # of crevice samples, c= # of U-bend samples, d = # of witness samples for surface analysis.

2.2.10 Task 10: Aging and Phase Stability of Alloy 22

The objectives of this task are to improve confidence in phase stability models and predictions; improve understanding of the influence of aging time, temperature and metallurgy on material phase stability; and provide data to performance margin analysis activities where appropriate.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the analysis developed in *Aging and Phase Stability of Waste Package Outer Barrier,* (BSC 2004 [DIRS 171924]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Samples for various heats of Alloy 22 with Cr content as high as 21.94% and Mo content as high as 14.00% have been aged at several temperatures for various times (approaching 1000 hours) and are available for analysis (see Table 2.2.10.1). The temperatures used in the aging studies include: 593ºC, 649ºC, 704ºC and 760ºC. The total number of sample variations available for testing is 72 (3 heats * 4 aging temperatures * 6 aging times). EBSD will be used to calculate the volume fraction of phases in these specimens and the results compared with the data in ANL-EBS-MD-000002 to determine if the chemistry range can be expanded. Calphad software will be used to determine the thermodynamic stability of structures found from analysis of the aged samples. Table 2.2.10.2 summarizes the FY08 testing for this task.

Alloys C-4, C-276 and G-30 have also been aged for 100,000 hours at at 260°C, 343°C and 427°C. Comparing the aging and phase stability of these other Ni-Cr-Mo alloys to Alloy 22 will improve our understanding and defensibility of the aging and phase stability model. These analyses will be performed in FY09 (Table 2.2.10.3).

The thermal aging facility at LLNL contains welded samples introduced into the furnaces in March 2005. The longest initial thermal aging time varied from 1,000 hours at the highest temperature (750°C) to 20 years at the lowest temperature (200°C). The aging temperatures are: 750°C, 700°C, 650°C, 600°C, 550°C, 500°C, 450°C, 400°C, 300°C, and 200°C. The last removal of specimens was in May 2006 for samples aged for 10,000 hours. The aging temperatures for the 10,000-hr samples are: 450°C, 500°C, 550°C, 600°C, 650°C, and 700°C. These samples will be analyzed for secondary phase precipitation in FY10 (Table 2.2.10.4).

Out year activities will involve improving the fidelity of the description for long-term phase stability of Alloy 22 and examining the effects on phase stability of both major and minor alloying elements such as chromium, molybdenum, iron and tungsten (Table 2.2.10.5).

Table 2.2.10.1. Task 10, FY08 Matrix 1: Comparison of aged Alloy 22 compositions with the current model range and the ASTM range.

Heat or Composition Range Designation	Cr%	Mo%	W%	Fe%
ASTM Range	20 to 22.5	12.5 to 14.5	2.5 to 3.5	Max. 6
Current Model Range	20.0 to 21.4	12.5 to 13.5	2.5 to 3.0	2 to 4.5
2277-6-3181	21.94	13.51	3.02	3.52
2277-9-3201	21.90	14.00	2.80	4.50
2277-3-3223	21.77	13.44	2.99	3.86

Table 2.2.10.2. Task 10, FY08 Matrix 2: Summary of calculations and sample characterization to be performed in FY08.

Type of Test	EBSD[1]	Calphad – ThermoCalc[2]
Number of Samples (Tests)	72	3
TBD Tests	18	9

[1]To measure second phase precipitate volume.
[2]To determine thermodynamic stability.

EBSD = electron backscatter diffraction.

Table 2.2.10.3. Task 10, FY09 Matrix 1: Analysis of Ni-Cr-Mo samples aged for 105 hours.

Type of Test	EBSD[1]	Calphad – ThermoCalc[2]
Number of samples (tests)	24[3]	12

[1]To measure second phase precipitate volume. EBSD = electron backscatter diffraction.
[2]To determine thermodynamic stability.
[3] 4 alloys x 2 metallurgical conditions (welded, non-welded) x 3 temperatures=24 sample variations.

Table 2.2.10.4. Task 10, FY10 Matrix 1: Analysis of Alloy 22 samples aged for 104 hours.

Type of Test	EBSD[1]	Calphad – ThermoCalc[2]
Number of Samples (Tests)	12 [3]	12
TBD Tests[4]	6	3

[1]To measure second phase precipitate volume. EBSD = electron backscatter diffraction.
[2]To determine thermodynamic stability.
[3] 6 temperatures x 1 aging time (10^4 hours) x 2 metallurgical conditions (welded, non welded) = 12 sample variations
[4] TBD tests may include the determination of second phase for Alloy 59 (N06059) to compare its phase stability relatively to Alloy 22.

Table 2.2.10.5. Task 10, FY11-17 Matrix 1: Summary of out year activities for aging and phase stability of Alloy 22.

Goal	Notes	Approximate number of Samples	Approximate Duration
Determine the long term phase stability of Alloy 22 at lower temperatures (400°C and below). Determine the viability of the extrapolation of the TTT diagram for longer times and lower temperatures.	Examine samples of Alloy 22 aged at 200°C, 300°C and 400°C for 10-years (available from LLNL aging facility in March 2015). Also examine second phase precipitation at the higher temperatures for times shorter than 10 years (e.g., 5,000 and 50,000 hours for intermediate temperatures). Perform correlations between type and amount of precipitates and the microchemistry of different heats of Alloy 22.	120 Tests (EBSD microscopy on samples and Calphad and ThermoCalc modeling calculations, including replicates)	5 years
Analyze the thermal stability behavior of other commercial Ni alloys and compare with Alloy 22 to determine the influence of major alloying elements such as chromium (Cr) and molybdenum (Mo). Also determine the effect of minor alloying elements such as iron (Fe) and tungsten (W). Correlate phase stability with the presence of impurities such as Si, P, S and Mn.	In order to improve confidence in the long term thermal stability of Alloy 22, it is important to examine the thermal stability of other Ni-based engineering alloys which contain different proportions of the important alloying elements such as Cr, Mo and W. For example, examination of Alloy 59 will determine the effect of residual Fe and the impact of W. Assess the predictive capabilities of Calphad and ThermoCalc softwares.	108 tests (including samples and calculations) (3 alloys, 3 temperatures, 3 time steps, 3 replicates)	2 years

2.2.11 Task 11: Stress Corrosion Cracking

The objectives of this task are to improve confidence in SCC initiation and propagation models and predictions; improve understanding of the influence of chemical environment, stress state and exposure time on SCC initiation and damage accumulation (if cracking is observed); and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the analysis developed in *Stress Corrosion Cracking of Waste Package Outer Barrier and Drip Shield Materials*, (SNL 2007 [DIRS 181953]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Due to the ongoing, multi-year nature of this testing, the activity descriptions are grouped by fiscal year with sub-headings for individual tasks.

Constant Load Tests (FY08)

Very long-term (~32,000 hours) constant load Keno tests are being carried out in an aerated, 105ºC, 15% basic saturated water (BSW) seepage type brine (Table 2.2.11.1). Additional Titanium Grade 28 and Grade 29 specimens including notched specimens will be added in FY08 (shown in grey shading in Table 2.2.11.1). For the titanium alloy specimens stress levels will be below the levels at which creep rupture is expected to occur for at least one year. To confirm that titanium alloy brine failures resulted from SCC rather than creep rupture, the ongoing series of creep tests (Table 2.2.11.2) will be completed in FY08 with 8 new specimens tested (shown in grey shading). Sufficient applied stress levels will be tested for each titanium alloy to enable measurement of both the steady state creep rate and the rupture time over the range of relevant applied stresses. Selected fractography and metallography will be performed on both Alloy 22 and the titanium alloy specimens to establish the cracking/fracture modes to help distinguish between SCC and creep rupture.

U-bend SCC Initiation Tests (FY08)

Ongoing (~15,000 hours) single and double U-bend tests listed in Tables 2.2.11.3 and 2.2.11.4, respectively will be continued in 165ºC aerated SCW brine along with additional Titanium alloy specimens that will replace some of the selected current Alloy 22 replicate specimens. This brine can be defended as a 'worst-case' seepage type brine for Alloy 22 and the drip shield titanium alloys based on existing project and literature results (see, ANL-EBS-MD-000005). If facilities become available, a second U-bend test campaign will be initiated in a deliquescent brine environment. The brine composition will be one of the test environments listed in Task 11, FY08 Matrix 6 or one selected based on the currently underway deliquescent brine evaluation. The matrix will include Alloy 22 and Titanium Grades 7, 28, and 29 single and double U-bend specimens.

Simulated Weld Flaw SCC Initiation Tests (FY08)

Since the waste package outer barrier final closure lid weld is stress mitigated to a depth of about 4 mm, the most likely SCC initiation sites are potential weld flaws. To simulate such flaws, a series of fatigue pre-cracked compact tension specimens covering a range of stress intensity

factor values have been on test in an aerated 105ºC, 15% BSW brine for about 5,000 hours (Table 2.2.11.5). More recently, Titanium Grades 28 and 29 specimens were added to the test matrix.

Low Temperature Threshold for SCC in Alloy 22 (FY08)

An additional FY08 task will be to determine if a lower temperature threshold exists below which SCC in Alloy 22 will not initiate and/or propagate under relevant brine test conditions. A reversing DC crack growth rate measurement system will be used to provide a relatively rapid approach to evaluating the lower temperature limit below which SCC will potentially not initiate and/or propagate in Alloy 22. SCC will be initiated in fatigue pre-cracked compact tension specimens exposed in brine-containing autoclaves by use of cyclic loading followed by increasing hold times at maximum load. Using, for example, a one-hour hold time, a growth rate can be established at a temperature at which SCC crack growth was previously observed. Then, the temperature will be lowered in step increments and a new rate established at each lower temperature. Using this sequential approach, the temperature below which crack growth is not observed may be obtained in approximately a few thousand hours for each environment evaluated. This approach will be applied to SCW brine since it is among the most SCC aggressive compositions for Alloy 22. Initial evaluation will be performed on an as-welded compact tension specimen. While at least three specimens will be studied, a test matrix cannot be developed at this time as this experiment is an iterative process. Additional environments and titanium crack growth rate specimens may be studied if facilities become available and time permits.

Electrochemical Testing and Passive Film Characterization to Support SCC and HIC Models (FY08)

Initial electrochemical tests for both Alloy 22 and Titanium Grades 7 and 29 are underway in several seepage and deliquescent type brine environments described in Table 2.2.11.6. Tests to date have determined the corrosion rate ratios for Titanium Grade 29/Titanium Grade 7 and have evaluated hydrogen pickup parameters, this work will be completed in FY08. In addition, the long term E_{CORR} and corrosion rates are being monitored and are planned for continuing monitoring in FY08 for both Alloy 22 and Titanium Grade 7 (with new specimens of Titanium Grade 28 and 29 to be added) in aerated BSW 95ºC brine with the test duration currently at about 6 years. In addition, the passive films on both alloys have been characterized after up to 5 years on test. These TEM and other film characterization tests will be continued in FY08 to confirm that the film structure remains stable and to quantify film thickness over time.

Constant Load Tests (FY10/11)

To increase defensibility, the constant load tests that are currently underway for Alloy 22 and titanium alloys (Titanium Grades 7, 28, and 29) for smooth and notched specimens will be continued in FY10/11. A new series of constant-load tests will be initiated to evaluate the weldment combinations planned for the drip shield. These include the following combinations: Titanium Grade 7/Titanium7, Titanium Grade 29/Titanium Grade 29, Titanium Grade 29/Titanium Grade 28/Titanium Grade 7. The SCC behavior of these similar and dissimilar material combinations in a repository relevant environment will be evaluated. The test matrix is shown in Table 2.2.11.7.

U-Bend SCC Initiation Tests (FY10/11)

The single and double U-bend tests underway in FY 2008 will continue, while new U-bend tests in one other test environment (to be determined) will be initiated (Tables 2.2.11.8 and 2.2.11.9). This will require two complete high temperature and high pressure air purged systems.

Weld Defect Tolerance Testing (FY10/11)

To evaluate the potential for SCC initiation at waste package weld flaws, the tests underway in 2008 will be continued to obtain longer times on test to improve confidence in results obtained to date. In addition, specimens representing the various titanium alloy weldment combinations in the drip shield will be added to the test matrix to evaluate the effect of weld flaws in titanium weldments. The ongoing and added specimens and test conditions are shown in Table 2.2.11.10.

Low Temperature Threshold for SCC in Alloy 22 (FY10/11)

Utilizing the testing approach initiated in FY08, any ongoing tests in aerated 150°C SCW brine and other selected brines will be completed and a series of new tests in two or more alternate brines (TBD based on the FY08 results) will be performed in FY09/10 to strengthen the technical basis for establishing a threshold temperature for SCC growth. Testing will be performed on both welded Alloy 22 fatigue pre-cracked compact tension specimens as well as on similar type specimens fabricated from dissimilar metal welded drip shield titanium alloys.

Electrochemical Testing and Passive Film Characterization to Support SCC and HIC Models (FY10/11)

In FY09/10 long-term electrochemical and weight loss measurements as well as hydrogen pickup measurement will be performed on Alloy 22 and Titanium Grade 7, 28, and 29 in selected environments (TBD based on FY08 shorter term tests) with temperatures up to about 220°C. Several potential test environments include SCW plus the brines shown in Table 2.2.11.11. In addition, the currently ongoing long-term E_{CORR} and hydrogen pickup tests in aerated 95°C BSW brine will be completed in FY09/10 after about 9 years total exposure. To improve confidence in the proposition that the passive films on Alloy 22 and drip shield titanium alloys remain stable over long periods after exposure in repository relevant environments, select passive film characterization tests including TEM and SIMS tests will be completed. Specimens will include one or more of the FY08 exposed specimens as well two of the longer-term specimens exposed during FY09/10. Also, one each of the very long-term (~8 to 9 years) Alloy 22 and Titanium Grade 7 specimens exposed in BSW brines will be characterized to supplement the currently available one year to five year results. This activity is currently assigned to the SCC task as the work in these areas is ongoing and interdependent. However, the majority of oxide characterization work for FY08 through FY17 will be carried out under *Task 7: Characterization of Alloy 22 and Titanium Oxides*. The two oxide characterization efforts will complement one-another.

Long-term U-bend testing on baseline materials (FY11-17)

Long-term exposure testing will be performed on double U-bend samples of the baseline materials for the waste package outer barrier and drip shield including Alloy 22, Titanium Grade 7 and Titanium Grade 29 (Table 2.2.11.12). The environments are selected to evaluate the effects on SCC behavior of the anticipated primary variables of T, chloride concentration and nitrate concentration and the anticipated secondary variables of lead, fluoride, bicarbonate and silicate. The selection of TBD environments will be based primarily on results and analyses

from testing performed during FY08 through FY10. The listed environments may also be modified if further analysis indicates that a different selection of environments, but the same approximate total number, would provide more complete information or information that better targets a specific corrosion concern.

Long-term U-bend testing on thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (FY11-17)

Long term exposure testing will be performed on double U-bend samples of thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (Table 2.2.11.13). These tests will determine any differences between the behavior observed for baseline materials (Table 2.2.11.12) and other mechanical or thermal conditions, other pretreatments (e.g., black anneal or deliquescent pre-exposure) and galvanic coupling. These tests will also provide information on how lower alloyed nickel-chromium-molybdenum materials behave compared to Alloy 22 and how potential surrogate titanium alloys (TBD) behave compared to Titanium Grades 7 and 29.

Long-term CT-sample testing (FY11-17)

The crack initiation behavior of barrier materials and surrogates in the presence of flaws will be evaluated by long-term testing of pre-cracked compact tension (CT) type specimens (Table 2.2.11.14). A potential drop technique will be used in-situ to intermittently check for crack advance on each sample. Post test SEM will also be used to examine fracture surface for indications of SCC. Samples will likely be loaded using a fixed weight or other suitable approach.

Constant load testing of Alloy 22 and titanium alloys (FY11-17)

Alloy 22 and titanium alloys specimens plus selected surrogate specimens at $\leq 220^{o}$C will be tested under constant load conditions for long-durations. The available equipment through FY10 is capable of 185 samples at a time. The samples on this machine will be removed and replenished as appropriate with most if not all replaced by FY12. A second Keno rig will be built capable of testing more aggressive environments and at higher temperatures. This second Keno rig will be loaded with a TBD environment and another 185 samples. Therefore a total of ~370 additional samples not previously initiated will be tested in this timeframe.

Table 2.2.11.1. Task 11, FY08 Matrix 1: Constant load Keno tests in aerated, 105ºC, 15% BSW seepage type brine.

Material	Condition	Finish	Specimens per Condition			Total Specimens
			System Pressure = 1,500 psi			
Alloy-22		RMS	85 ksi	93 ksi	100 ksi	
	As-received	150	6	6	6	18
	As-received	72		6		6
	AR + HT1 (700°C/175 hr)	150	6	4		10
	AR + HT2 (520°C/1,000 hr)	150	6	6		12
	AR + 20%CW + HT2			6		6
	20% Cold worked			6		6
	AR + Creviced	150	6	6		12
	AR + HT1 + Creviced	150	6	6		12
	Weld and HAZ	150	6	6		12
Alloy 22 notched	As-received	150		8		8
Alloy 22 notched	As-received + 700°C/175 hr	150		8		8
Alloy 22 notched	Weld and HAZ	150		8		8
Titanium Grade 7			26 ksi	31 ksi		
	As-received	150	4	4		8
Titanium Grade 29			69 ksi	90 ksi	99 ksi	
	As-received	150	4	3	1	8
Titanium Grade 29 notched**			TBD	TBD		
	As-received	150	4	4		8
Titanium Grade 28**			TBD	TBD		
	As-received	150	4	4		8
Titanium Grade 28 notched**			TBD	TBD		
	As-received	150	4	4		8
NG 316 SS			65 ksi	70 ksi		
	As-received	150	1	6		7
	Creviced	150		6		6

** In FY08, 24 specimens as noted above will be added along with 20 additional titanium alloy specimens to be determined. It may be necessary to replace a limited number of redundant Alloy 22 specimens currently on test.

Table 2.2.11.2. Task 11, FY08 Matrix 2: Creep testing of Titanium Grades 7, 28 and 29.

Specimen	Material*	Temperature	Stress	Creep Rate, s^{-1}
Ti7c1	Titanium Gr. 7	105°C	45 ksi	7.7×10^{-6} (10.1)**
Ti7c2	Titanium Gr. 7	105°C	50.7 ksi	4.0×10^{-5} (1.64)**
Ti7c3	Titanium Gr. 7	105°C	47.5 ksi	3.3×10^{-5} (6.2)**
Ti7c4	Titanium Gr. 7	105°C	40 ksi	1.5×10^{-7} (493)**
Ti7c5	Titanium Gr. 7	105°C	47.25 ksi	7.7×10^{-5} (1.17)**
Ti7c6	Titanium Gr. 7	105°C	41.3 ksi	4.6×10^{-7} (143)**
Ti7c7	Titanium Gr. 7 CW	105°C	68 ksi	6.6×10^{-7} (11.2)**
Ti7c8	Titanium Gr. 7 CW	105°C	56.7 ksi	1.7×10^{-9} (224)**
Ti7c8	Titanium Gr. 7 CW	105°C	60.5 ksi	7.6×10^{-10} (224)**
Ti7c8	Titanium Gr. 7 CW	105°C	64.3 ksi	1.4×10^{-8} (224)**
Ti29c1	Titanium Gr. 29	150°C	89.25 ksi	1.8×10^{-9}
Ti29c2	Titanium Gr. 29	150°C	94.5 ksi	8.9×10^{-10}
Ti28c2	Titanium Gr. 28	150°C	64.8 ksi	5.5×10^{-10}
Ti28c2	Titanium Gr. 28	150°C	72 ksi	1.9×10^{-6}
2 of	Titanium Gr. 7	TBD	TBD	
3 of	Titanium Gr. 28	TBD	TBD	
3 of	Titanium Gr 29	TBD	TBD	

* Titanium Gr. 7 CW = 20% reduction in thickness by forging at 25°C.

** Specimen failed at the hours indicated in parentheses.

NOTE: In FY08, ongoing tests will be completed, and 8 new tests initiated.

Table 2.2.11.3. Task 11, FY08 Matrix 3: Single U-bend testing in 165ºC aerated SCW brine.

Identification	Heat	Condition*
C22 (227793263) TCP No. 1	2277-9-3263	TCP No. 1
C22 (227793263) LRO No. 1	2277-9-3263	LRO No. 1
Titanium Grade 29 (956205) AR No. 1**	956205	AR No. 1
Titanium Grade 28 AR**	TBD	TBD
C22 (227793263) LRO No. 2	2277-9-3263	LRO No. 2
C22 (227793263) AR No. 2	2277-9-3263	AR No. 2
Titanium Grade 29 (956205) AR No. 2**	956205	AR No. 2
Titanium Grade 28 AR**	TBD	TBD
C22 (227793263) AR No. 3	2277-9-3263	AR No. 3
C22 (227793263) TCP No. 4	2277-9-3263	TCP No. 4
C22 (227793263) LRO No. 4	2277-9-3263	LRO No. 4
C22 (227793263) AR No. 4	2277-9-3263	AR No. 4
C22 (227793263) TCP No. 5	2277-9-3263	TCP No. 5
C22 (227793263) LRO No. 5	2277-9-3263	LRO No. 5
C22 (227793263) AR No. 5	2277-9-3263	AR No. 5
Titanium Grade 28 AR**	TBD	TBD
Titanium Grade 28 AR**	TBD	TBD
C22 (227793263) AR No. 6	2277-9-3263	AR No. 6
Titanium Grade 29 (956205) AR No. 3	956205	AR No. 3
Titanium Grade 29 (956205) AR No. 4	956205	AR No. 4

* Machined U-bends after heat treatment below.

**To be added in FY08. Additional replacement of a limited number of replicate Alloy 22 specimens with titanium alloys may also be implemented in FY08.

AR = as-received (as-welded).

TCP (topologically close packed) = 650°C for 200 hrs with water quench.

LRO (long range ordering) = 550°C for 10 hrs with water quench.

Table 2.2.11.4. Task 11, FY08 Matrix 4: Double U-Bend testing in 165ºC aerated SCW brine.

Identification	Heat	Condition	Arm Spread Before Test (in)	Arm Spread After Test* (in)
DUB1181(outer) / DUB1061(inner)	2277-9-3241	As-received	0.998	0.975
DUB1182(outer) / DUB1062(inner)	2277-9-3241	As-received	1.009	0.974
DUB1183(outer) / DUB1063(inner)	2277-9-3241	As-received	1.009	0.977
DUB1184(outer) / DUB1064(inner)	2277-9-3241	As-received	1.010	0.974
DUB1185(outer) / DUB1065(inner)	2277-9-3241	As-received	1.004	0.973
DUB1186(outer) / DUB1066(inner)	2277-9-3241	As-received	1.005	0.975
DUB1187(outer) / DUB1067(inner)	2277-9-3241	As-received	1.007	0.970
DUB1188(outer) / DUB1068(inner)	2277-9-3241	As-received	1.005	0.973
DUB1189(outer) / DUB1069(inner)	2277-9-3241	As-received	1.009	0.975
DUB1190(outer) / DUB1070(inner)	2277-9-3241	As-received	1.006	0.972

* Dimensions shown for inspection after 3,431 hours; then, all U-bends were retightened as shown to compensate for possible stress relaxation.

Table 2.2.11.5. Task 11, FY08 Matrix 5: Weld defect tolerance specimens in 105ºC, 15% BSW seepage type brine.

CTS	Material and Condition	Stress Intensity Factor
#1	C22 As-welded (AW)	higher K (30 ksi√in)
#2	C22 As-welded (AW)	lower K (20 ksi√in)
#3	C22 As-welded (AW)	lower K (20 ksi√in)
#4	C22 As-welded (AW)	higher K (30 ksi√in)
#5	Titanium Grade 29 (As received)	higher K (30 ksi√in)
#6	Titanium Grade 29 (As received)	lower K (20 ksi√in)
#7	C22 AW+TCP	higher K (30 ksi√in)
#8	C22 AW+TCP	lower K (20 ksi√in)
#9	C22 AW+SHT + Water quenched	higher K (30 ksi√in)
#10	C22 AW+SHT + Water quenched	lower K (20 ksi√in)
#11	C22 AW+SHT + Water quenched	higher K (30 ksi√in)
#12	C22 AW+SHT + Water quenched	lower K (20 ksi√in)
#13	Titanium Gr 28 (As received)	lower K (20 ksi√in)
#14	C22 AW +SHT +Air blasted	higher K (30 ksi√in)
#15	Titanium Gr 28 (As received)	higher K (30 ksi√in)
#16	C22 AW + SHT + Still air cool	lower K (20 ksi√in)
#17	C22 Base	lower K (20 ksi√in)
#18	C22 Base	higher K (30 ksi√in)

SHT = 1,120°C / 30 minutes / water quench unless otherwise specified.
As-welded = precrack in weld metal.
AW + TCP = precrack in weld metal, heat treat at 700°C/ 75 hours.
AW + SHT with water quench, precrack in weld metal.
AW + SHT with still air cool down, precrack in weld metal.
AW + SHT with air blast cool down, precrack in weld metal.
As-received material, fatigue crack not in weld metal.

Table 2.2.11.6. Task 11, FY08 Matrix 6: Electrolyte compositions for electrochemical testing of Alloy 22 and titanium alloys.

ID	Temp. (°C)	pH	$CaCl_2$	KCl	KNO_3	$NaNO_3$	NaCl	$Na2SO_4$	NaF	NaBr
TS-1	150	4.9	3.6	5.8	1.8	2	0	0	0	0
TS-2	120	7.3	0	7	0	1.8	1.2	2.4	0	0
TS-3	120	9.0	0	7.2	0.3	3.3	0	2.1	0.2	0
TS-4	150	9.7	0	5	2.8	6.6	0	6.8	0.1	0.1
TS-5	120	4.9	3.6	5.8	1.8	2	0	0	0	0
TS-6	180	—	—	—	50	50	—	—	—	—
TS-7	180	—	—	2	50	50	2	—	—	—
TS-8	220	—	—	—	50	50	—	—	—	—
TS-9	220	—	—	2	50	50	2	—	—	—

NOTE: Concentrations are in molal.

Table 2.2.11.7. Task 11, FY09/10 Matrix 1: Constant load testing.

Material	Condition	Specimens per Condition			Total Specimens
		System Pressure = 1,500 psi			
Alloy-22		85 ksi	93 ksi	100 ksi	
	As-received			3	3
	20% Cold worked		3		3
	Weld and HAZ		3		3
Titanium-Grade 7, Smooth Specimen		26 ksi	31 ksi	TBD	
	As-received	4*	4*	4	8*+4
	As-Welded Plus SR*	4	4	4	12
Titanium Grade 7 Notched		TBD	TBD	TBD	
	Notch in SR Weld Metal	4	4	4	12
	Notch in SR HAZ	4	4	4	12
	Notch in Base Metal	4	4	4	12
Titanium Grade 29, Smooth Specimen		69 ksi	90 ksi	99 ksi	
	As-received	4*	3*	1*	8*
	As-Welded Plus SR*	4	4	4	12
Titanium Grade 29 Notched		TBD	TBD	TBD	
	Notch in SR Weld Metal	4	4	4	12
	Notch in SR HAZ	4	4	4	12
	Notch in Base Metal	4	4	4	12
Titanium-Grade 7/Titanium Grade 28/Titanium Grade 29, Smooth Specimen		26 ksi	31 ksi	TBD	
	As-welded Plus SR*	4	4	4	12
Titanium-Grade 7/Titanium Grade 28/Titanium Grade 29 Notched		TBD	TBD	TBD	
	Notch in SR Ti7/Ti28 Weld Metal	4	4	4	12
	Notch in SR Ti29/Ti28 Weld Metal	4	4	4	12
	Notch in SR Ti7/Ti28 HAZ	4	4	4	12
	Notch in SR Ti29/Ti28 HAZ	4	4	4	12
Total Specimen Number: 185 + 30 potential replacement specimens with TBD test conditions.					

NOTE: SR-stress relieved.

* Specimens currently on test (started in FY08 or earlier).

Table 2.2.11.8. Task 11, FY09/10 Matrix 2: U-Bend Tests.

Material and Condition	Number of Specimens
Single U-Bend	
As-Welded Plus SR Titanium Grade 7	4
As-Welded Plus SR Titanium Grade 29	4
As-Welded Plus SR Titanium-Grade 7/Titanium Grade 28/Titanium Grade 29	4

Table 2.2.11.9. Task 11, FY09/10 Matrix 3: Double U-Bend Tests.

Double U-Bend	
Titanium Alloys	
As-Welded Plus SR Titanium Grade 7	4
As-Welded Plus SR Titanium Grade 29	4
As-Welded Plus SR Titanium-Grade 7/Titanium Grade 28/Titanium Grade 29	4
Alloy 22	
As-welded Alloy 22	2
Cold worked Alloy 22	2
Plasticity burnished Alloy 22 weld	2

Table 2.2.11.10. Task 11, FY09/10 Matrix 4: Weld Defect Tolerance Specimens.

CTS	Material and Condition	Stress Intensity Factor
FY08 Carried Over Specimens		
#1	C22 As-welded (AW)	30 ksi√in
#5	Titanium Grade 29 (As received)	30 ksi√in
#7	C22 AW+TCP	30 ksi√in
#9	C22 AW+SHT + Water quenched	30 ksi√in
#14	C22 AW +SHT +Air blasted	30 ksi√in
#15	Titanium Grade 28 (As received)	30 ksi√in
#18	C22 Base	30 ksi√in
New Specimens		
TBD	Ti7/Ti28/Ti29 As-Welded Plus SR	30 ksi√in
TBD	Ti7/Ti28/Ti29 As-Welded Plus SR	30 ksi√in
TBD	Ti29/Ti29 As-Welded Plus SR	30 ksi√in
TBD	Ti29/Ti29 As-Welded Plus SR	30 ksi√in
TBD	Ti7/Ti28/Ti29 As-Welded	30 ksi√in
TBD	Ti7/Ti28/Ti29 As-Welded	30 ksi√in
TBD	Ti29/Ti29 As-Welded	30 ksi√in
TBD	Ti29/Ti29 As-Welded	30 ksi√in
TBD	Ti7/Ti28/Ti29 As-Welded	35 ksi√in
TBD	Ti29/Ti29 As-Welded	35 ksi√in
TBD	Ti28/Ti29 As-Welded	35 ksi√in

NOTE: For the welded specimens, the fatigue pre-crack tips are in weld metal.

Table 2.2.11.11. Task 11, FY09/10 Matrix 5: Test environments for long-term electrochemical measurements.

ID	Temp. (°C)	pH	$CaCl_2$	KCl	KNO_3	$NaNO_3$	NaCl	$Na2SO_4$	NaF	NaBr
TS-1	150	4.9	3.6	5.8	1.8	2	0	0	0	0
TS-2	120	7.3	0	7	0	1.8	1.2	2.4	0	0
TS-3	120	9.0	0	7.2	0.3	3.3	0	2.1	0.2	0
TS-4	150	9.7	0	5	2.8	6.6	0	6.8	0.1	0.1
TS-5	120	4.9	3.6	5.8	1.8	2	0	0	0	0
TS-6	180	—	—	—	50	50	—	—	—	—
TS-7	180	—	—	2	50	50	2	—	—	—
TS-8	220	—	—	—	50	50	—	—	—	—
TS-9	220	—	—	2	50	50	2	—	—	—

NOTE: Concentrations are in molal.

Table 2.2.11.12. Task 11, FY11-17 Matrix 1: Rough order estimate of long-term double U-bend sample testing for Alloy 22, and Titanium Grades 7 and 29 (2,664 samples).

[Cl^-] / *m*	[NO_3^-] / *m*	Other composition	T (°C)	# Samples of each material analyzed at each time period								
				0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
1	0	-	90	—	—	—	3	3	3	5	5	5
10	0	-	60	—	—	—	3	3	3	5	5	5
1	0.01	-	30	—	—	—	3	3	3	5	5	5
1	0.1	-	60	—	—	—	3	3	3	5	5	5
1	1	-	90	—	—	—	3	3	3	5	5	5
—	—	SAW	30	—	—	—	3	3	3	5	5	5
—	—	SAW	60	—	—	—	3	3	3	5	5	5
—	—	SAW	90	—	—	—	3	3	3	5	5	5
—	—	SDW	30	—	—	—	3	3	3	5	5	5
—	—	SDW	60	—	—	—	3	3	3	5	5	5
—	—	SDW	90	—	—	—	3	3	3	5	5	5
—	—	SCW	30	—	—	—	3	3	3	5	5	5
—	—	SCW	60	—	—	—	3	3	3	5	5	5
—	—	SCW	90	—	—	—	3	3	3	5	5	5
0	0	Distilled Water	90	—	—	—	3	3	3	5	5	5
1	0	1 ppm Pb	90	—	—	—	3	3	3	5	5	5
1	0	10 ppm Pb	90	—	—	—	3	3	3	5	5	5
1	0.1	10 ppm Pb	90	—	—	—	3	3	3	5	5	5
1	0	0.02 *m* F	90	—	—	—	3	3	3	5	5	5
1	0	0.2 *m* F	90	—	—	—	3	3	3	5	5	5
1	0.1	0.2 *m* F	90	—	—	—	3	3	3	5	5	5
1	0	0.02 *m* HCO_3^-	90	—	—	—	3	3	3	5	5	5
1	0	0.2 *m* HCO_3^-	90	—	—	—	3	3	3	5	5	5
1	0.1	0.2 *m* HCO_3^-	90	—	—	—	3	3	3	5	5	5
1	0	4 mg/l SiO_3^{2-}	90	—	—	—	3	3	3	5	5	5
1	0	40 mg/l SiO_3^{2-}	90	—	—	—	3	3	3	5	5	5
1	0.1	40 mg/l SiO_3^{2-}	90	—	—	—	3	3	3	5	5	5
—	—	TBD-1	—	—	—	—	3	3	3	5	5	5
—	—	TBD-2	—	—	—	—	3	3	3	5	5	5
—	—	TBD-3	—	—	—	—	3	3	3	5	5	5
—	—	TBD-4	—	—	—	—	3	3	3	5	5	5
—	—	TBD-5	—	—	—	—	3	3	3	5	5	5
—	—	TBD-6	—	—	—	—	3	3	3	5	5	5
—	—	TBD-7	—	—	—	—	3	3	3	5	5	5
—	—	TBD-8	—	—	—	—	3	3	3	5	5	5
—	—	TBD-9	—	—	—	—	3	3	3	5	5	5
—	—	TBD-10	—	—	—	—	3	3	3	5	5	5

Table 2.2.11.13. Task 11, FY11-17 Matrix 2: Rough order estimate of long-term double U-bend sample testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials (1324 samples).

Envrionments: **1 *m* NaCl + 0.1 *m* HCO_3, 90°C, neutral pH** **1 *m* NaCl + 0.1 *m* NaF, 90°C, neutral pH** **SAW, 90°C** **SCW, 90°C**										
		# Samples analyzed at each time period, per environment, per material								
Material	**Condition**	**0.1 yr**	**0.25 yr**	**0.5 yr**	**1 yr**	**2 yr**	**5 yr**	**10 yr**	**20 yr**	**Extra**
Alloy 22	Black Anneal	—	—	—	—	—	6	10	10	20
Alloy 22	LPB	—	—	—	—	—	3	5	5	5
Alloy 22	Simulated weld root	—	—	—	—	—	3	5	5	5
Alloy 22	Simulated HAZ	—	—	—	—	—	3	5	5	5
C276	Annealed	—	—	—	—	—	3	5	5	5
Ni	Annealed	—	—	—	—	—	3	5	5	5
Nichrome 80-20	Annealed	—	—	—	—	—	3	5	5	5
Ni-20 Cr-7 Mo	Annealed	—	—	—	—	—	3	5	5	5
Ni-15 Cr-7 Mo	Annealed	—	—	—	—	—	3	5	5	5
316 SS	Annealed	—	—	—	—	—	3	5	5	5
Future material	TBD	—	—	—	—	—	3	5	5	5
Titanium Gr7/28/29	Weld	—	—	—	—	—	3	5	5	5
Titanium Grade 28	Annealed	—	—	—	—	—	3	5	5	5
TBD-1	—	—	—	—	—	—	3	5	5	5
TBD-2	—	—	—	—	—	—	3	5	5	5
TBD-3	—	—	—	—	—	—	3	5	5	5
TBD-4	—	—	—	—	—	—	3	5	5	5
TBD-5	—	—	—	—	—	—	3	5	5	5

Table 2.2.11.14. Task 11, FY11-17 Matrix 3: Rough order estimate of long-term CT-sample testing for Alloy 22, Alloy 22 surrogates, Titanium Grade 7 and Titanium Grade 29 (60 samples).

Material	Environment[(1)]	T / ºC	Stress Intensity	# Samples[1]
Alloy 22	SCW	90	TBD	3
	1 *m* NaCl + 0.1 *m* HCO_3	90	TBD	3
	1 *m* NaCl + 0.1 *m* NaF	90	TBD	3
C276	SCW	90	TBD	3
	1 *m* NaCl + 0.1 *m* HCO_3	90	TBD	3
	1 *m* NaCl + 0.1 *m* NaF	90	TBD	3
Ni-15 Cr-7 Mo	SCW	90	TBD	3
	1 *m* NaCl + 0.1 *m* HCO_3	90	TBD	3
	1 *m* NaCl + 0.1 *m* NaF	90	TBD	3
Titanium Grade 7	SCW	90	TBD	3
	1 *m* NaCl + 0.1 *m* HCO_3	90	TBD	3
	1 *m* NaCl + 0.1 *m* NaF	90	TBD	3
Titanium Grade 29	SCW	90	TBD	3
	1 *m* NaCl + 0.1 *m* HCO_3	90	TBD	3
	1 *m* NaCl + 0.1 *m* NaF	90	TBD	3

[1] One environment will be selected for alternate immersion testing in addition to the constant immersion testing. The alternate immersion testing will require 3 samples per material for a total of 15 additional samples.

2.2.12 Task 12: Analysis of U-bend Samples from the LTCTF

The objective of this task is to determine if SCC has initiated on U-bend samples exposed for 6.5 or 9.5 years in the LTCTF to improve confidence in the SCC resistance of Alloy 22 and titanium alloys (*Stress Corrosion Cracking of Waste Package Outer Barrier and Drip Shield Materials,* (SNL 2007 [DIRS 181953])).

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the analysis developed in *Stress Corrosion Cracking of Waste Package Outer Barrier and Drip Shield Materials*, (SNL 2007 [DIRS 181953]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

The archived U-bend samples from the LTCTF will be analyzed and documented for physical appearance including evidence of stress corrosion cracking or other localized corrosion. Tables 2.2.12.1 through 2.2.12.3 list the specimens that will be analyzed in FY08 for Alloy 22, Titanium Grade 7 and Titanium Grade 16, respectively. Tables 2.2.12.4 through 2.2.12.6 show the total number of specimens available for analysis, the selected samples to be analyzed in FY08 and the remaining samples to be analyzed in FY09. There are some redundancies in these two sets of Tables but all six tables are included in this document to maintain consistency with other predecessor planning documents. The analyses of the U-bend specimens will include optical microscopy of the sample surfaces and select samples will be imaged using SEM as appropriate. In FY08 a subset of the Alloy 22 samples from what are believed to be the most aggressive exposure environments will be examined; the remaining Alloy 22 specimens and the Titanium Grade 7 and Grade 16 specimens will be examined in FY09.

Table 2.2.12.1. Task 12, FY08 Matrix 1: Examination of select 9.5 year Alloy 22 U-bend specimens.

Electrolyte	T (°C)	Phase	Number of Samples Available[1]	Number of Samples to be Examined for SCC in FY08[1,2]
SDW	60	Aqueous	2	—
		Vapor	2	—
	90	Aqueous	2	2
		Vapor	2	—
SAW	60	Aqueous	12	—
		Vapor	12	—
	90	Aqueous	6	6[3]
		Vapor	6	—
SCW	60	Aqueous	6	6[3]
		Vapor	6	—
	90	Aqueous	6	6[3]
		Vapor	6	—

[1] The samples selected for examination in FY08 are considered to be from the most aggressive conditions. The observation of no SCC in these samples would be a strong indication that SCC has not occurred in the other conditions. The remainder of the samples will be examined in out-years.

[2] Welded and non-welded samples will be represented in the population of examined samples.

[3] Initially, only 5 of the 6 available samples will be analyzed for each exposure condition. It may be more beneficial to use the remaining sample for oxide characterization. This decision will be made based upon consideration of the relative surface finishes of the U-bend versus the crevice samples (Tasks 1 and 2) and/or on the outcome of the initial analysis for SCC.

Table 2.2.12.2. Task 12, FY08 Matrix 2: Examination of select 6.6 year Titanium Grade 7 U-bend specimens.

Electrolyte	T (°C)	Phase	Number of Samples Available[1]	Number of Samples to be Examined for SCC in FY08[1,2]
SDW	60	Aqueous	27	—
		Vapor	24	—
	90	Aqueous	24	6
		Vapor	24	—
SAW	60	Aqueous	24	—
		Vapor	24	—
	90	Aqueous	24	6
		Vapor	24	—
SCW	60	Aqueous	—	—
		Vapor	—	—
	90	Aqueous	48	12
		Vapor	48	—

[1] The samples selected for examination in FY08 are considered to be from the most aggressive conditions. The observation of no SCC in these samples would be a strong indication that SCC has not occurred in the other conditions. The remainder of the samples will be examined in out-years.

[2] Both welded and non-welded samples will be represented in the population of examined samples.

Table 2.2.12.3. Task 12, FY08 Matrix 3: Examination of select 9.5 year Titanium Grade 16 U-bend specimens.

Electrolyte	T (°C)	Phase	Number of Samples Available[1]	Number of Samples to be Examined for SCC in FY08[1,2]
SDW	60	Aqueous	1	—
		Vapor	1	—
	90	Aqueous	2	2
		Vapor	2	—
SAW	60	Aqueous	6	—
		Vapor	12	—
	90	Aqueous	6	6[3]
		Vapor	6	—
SCW	60	Aqueous	6	6[3]
		Vapor	6	—
	90	Aqueous	6	6[3]
		Vapor	6	—

[1] The samples selected for examination in FY08 are considered to be from the most aggressive conditions. The observation of no SCC in these samples would be a strong indication that SCC has not occurred in the other conditions. The remainder of the samples will be examined in out-years.

[2] Both welded and non-welded samples will be represented in the population of examined samples.

[3] Initially, only 5 of the 6 available samples will be analyzed for each exposure condition. It may be more beneficial to use the remaining sample for oxide characterization. This decision will be made based upon consideration of the relative surface finishes of the U-bend versus the crevice samples (Tasks 1 and 2) and/or on the outcome of the initial analysis for SCC.

Table 2.2.12.4. Task 12, FY09 Matrix 1: Examination of 9.5 year Alloy 22 U-bend specimens.

Electrolyte	T (ºC)	Phase	Total Number of Samples Available	Number of Samples to be Examined in FY08	Number of Samples to be Examined in FY09[1]
SDW	60	Aqueous	2	—	2
		Vapor	2	—	2
	90	Aqueous	2	2	—
		Vapor	2	—	2
SAW	60	Aqueous	12	—	12[2]
		Vapor	12	—	12[2]
	90	Aqueous	6	6	—
		Vapor	6	—	6[2]
SCW	60	Aqueous	6	6	—
		Vapor	6	—	6[2]
	90	Aqueous	6	6	—
		Vapor	6	—	6[2]

[1] Welded and non-welded samples will be represented in the population of examined samples.

[2] Initially, one sample from each condition will be withheld from cleaning and inspection. It may be more beneficial to use the remaining sample for oxide characterization. This decision will be made based upon consideration of the relative surface finishes of the U-bend versus the crevice samples (Tasks 1 and 2) and/or on the outcome of the initial analysis for SCC.

Table 2.2.12.5. Task 12, FY09 Matrix 2: Examination of 6.6 year Titanium Grade 7 U-bend specimens.

Electrolyte	T (ºC)	Phase	Total Number of Samples Available	Number of Samples to be Examined in FY08	Number of Samples to be Examined in FY09[1]
SDW	60	Aqueous	48	—	27[2]
		Vapor	24	—	24[2]
	90	Aqueous	24	6	18[2]
		Vapor	24	—	24[2]
SAW	60	Aqueous	24	—	24[2]
		Vapor	24	—	24[2]
	90	Aqueous	24	6	18[2]
		Vapor	24	—	24[2]
SCW	60	Aqueous	—	—	—
		Vapor	—	—	—
	90	Aqueous	48	12	36[2]
		Vapor	48	—	48[2]

[1] Welded and non-welded samples will be represented in the population of examined samples.

[2] Initially, one sample from each condition will be withheld from cleaning and inspection. It may be more beneficial to use the remaining sample for oxide characterization. This decision will be made based upon consideration of the relative surface finishes of the U-bend versus the crevice samples (Tasks 1 and 2) and/or on the outcome of the initial analysis for SCC.

Table 2.2.12.6. Task 12, FY09 Matrix 3: Examination of 9.5 year Titanium Grade 16 U-bend specimens.

Electrolyte	T (ºC)	Phase	Total Number of Samples Available	Number of Samples to be Examined in FY08	Number of Samples to be Examined in FY09[1]
SDW	60	Aqueous	1	—	1
		Vapor	1	—	1
	90	Aqueous	2	2	—
		Vapor	2	—	2
SAW	60	Aqueous	6	—	6[2]
		Vapor	12	—	12[2]
	90	Aqueous	6	6	—
		Vapor	6	—	6[2]
SCW	60	Aqueous	6	6	—
		Vapor	6	—	6[2]
	90	Aqueous	6	6	—
		Vapor	6	—	6[2]

[1] Welded and non-welded samples will be represented in the population of examined samples.

[2] Initially, one sample from each condition will be withheld from cleaning and inspection. It may be more beneficial to use the remaining sample for oxide characterization. This decision will be made based upon consideration of the relative surface finishes of the U-bend versus the crevice samples (Tasks 1 and 2) and/or on the outcome of the initial analysis for SCC.

2.2.13 Task 13: Hydrogen Embrittlement

The objectives of this task are to improve confidence in the modeling and prediction of hydrogen effects on engineered barrier materials; improve understanding of the influence of chemical environment and electrochemical conditions on hydrogen absorption and mechanical properties; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the analysis developed in *Stress Corrosion Cracking of Waste Package Outer Barrier and Drip Shield Materials*, (SNL 2007 [DIRS 181953]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Forced charging (either in a gas or aqueous phase) will be used to artificially introduce large quantities of hydrogen into titanium and Alloy 22 to assess the impact on mechanical properties (Table 2.2.13.1). Sample for hydrogen analysis and for mechanical property determination will be exposed for various durations to the charging environment. This approach will yield a relationship between total hydrogen content and degradation in mechanical properties.

For longer-term studies where a higher fidelity description of hydrogen effects will be obtained, it will be necessary to construct a thermal desorption spectroscopy (TDS) system. In this technique, the rate of hydrogen egress from a sample is monitored as a function of temperature and time. The resulting spectra provide information on the number of trap sites within the material, their relative strength, and (when combined with a microstructural analysis of the material) their effective coverage (or concentration, in the case of a trap site which is progressively generated by the charging process). By performing these experiments at a number of different thermal ramp rates, the migration energy for hydrogen within the metal lattice, as well as the binding energy to each trap site, can be determined.

Out year activities will target understanding the relationship between exposure to repository relevant electrolytes and the ensuing hydrogen uptake and impact on mechanical properties (Table 2.2.13.2). The hydrogen uptake characteristics of Alloy 22 and titanium will be assessed through a combination of open circuit exposures, exposures with impressed current or potential and short-term polarization experiments. Efforts will also be made to characterize the trap sites in these materials and develop an understanding of the electrochemical conditions necessary to cause hydrogen induced degradation.

Table 2.2.13.1. Task 13, FY10 Matrix 1: Establish relationship between bulk hydrogen content and mechanical properties for Titanium Grade 7 and Alloy 22.

Material	Environment	# Samples for Hydrogen Analysis[1,3], # Samples for Mechanical Testing[2,3]				
		Exposure Duration[4]				
		0 weeks (control)	1 week	2 weeks	4 weeks	8 weeks
Alloy 22	TBD[5]	5, 3	5, 3	5, 3	5, 3	5, 3
Titanium Grade 7	TBD[5]	5, 3	5, 3	5, 3	5, 3	5, 3
Titanium Grade 29	TBD[5]	5, 3	5, 3	5, 3	5, 3	5, 3

[1] Hydrogen analysis will be performed to measure bulk hydrogen content

[2] Mechanical properties will be determined by tensile testing. Samples will be dog-bone configuration with clamps used to secure sample in mechanical test frame.

[3] All samples will undergo a prescribed bake-out procedure to ensure that the hydrogen level starts at a controlled level and that meaningful changes in hydrogen concentration can be detected. Hydrogen will be introduced through an accelerated charging procedure, either electrochemical charging or gas phase charging.

[4] The duration of exposures may be modified based on the choice of charging technique and the rate at which hydrogen is expected to accumulate in the materials.

[5] The environment for hydrogen charging will be determined from a review of gas-phase and aqueous phase charging conditions in the literature.

Table 2.2.13.2. Task 13, FY11-17 Matrix 1: Rough order estimate for testing to support hydrogen embrittlement studies.

Goal	Notes	Approximate number of Samples	Approximate Duration
Establish relationship between bulk hydrogen content and mechanical properties for Titanium Grade 28	This activity completes the testing begun in Task 13, FY10, Matrix 1.	40 (One charging environment, durations of 0, 1, 2, 4, 8 weeks, 1 material, 5 H-content samples and 3-mechanical property samples per condition)	2 Years
Effect of long-term open circuit exposure on H content and mechanical properties	Testing will include Alloy 22, and Titanium grades 7, 28 and 29.	160 (One environment, Durations of 0, 5, 10, 15 and 20 years. 5 H-content samples and 3 mechanical property samples per duration)	20 years
Effect of long-term potentiostatic polarization on H content and mechanical properties	Materials will be polarized at 2 different potentials near the reversible potential for hydrogen. Testing will include Alloy 22, and Titanium grades 7, 28 and 29.	256 (One environment, 2 potentials, durations of 0, 0.5, 1 and 2 years, 4 materials, 5 H-content and 3 mechanical samples per duration)	3 years
Develop trap site identification	Combine TDS, varying charging conditions and metallurgical analysis to identify metallurgical features in TDS spectra.	75 (5 hydrogen concentrations, 3 grades of Titanium, 5 temperature ramp rates)	2 years
Establish relationship between electrochemical conditions at the metal surface and the rate of hydrogen ingress into the drip shield	Testing will include both Titanium Grade 7 and Titanium Grade 29. Devanathan-Stachurski permeation (or an analagous technique) experiments will be utilized to extract subsurface lattice hydrogen concentrations as a function of charging conditions. This will then be combined with TDS to establish trap site density and coverage as a function of charging current density, and then with mechanical testing to determine anticipated strength levels as a function of electrochemical surface conditions	100 (multiple charging conditions within a series of relevant environment chemistries)	2 years

2.2.14 Task 14: Corrosion Behavior of Neutron Absorber Materials

The objectives of this task are to improve confidence in general and localized corrosion predictions for neutron absorbing material; improve understanding of the influence of temperature, chemical environment, and exposure time on corrosion rate and propensity for localized corrosion for baseline and alternative materials; and provide data to performance margin analysis activities. Corrosion rates measured to date have relied on electrochemical testing over a relatively short immersion time – future testing will employ long term exposures and direct measurement of corrosion rate via weight loss.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the current treatment of corrosion of neutron absorber materials (*Evaluation of Neutron Absorber Materials Used for Criticality Control in Waste Packages* (BSC 2006 [DIRS 180664]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Long term exposure tests on 304-B SS will be performed to determine the average corrosion rate and the propensity for crevice corrosion initiation (Table 2.2.14.1). Samples will be removed at various time steps to determine how the corrosion rate depends on exposure time. Environments for testing will be defined based on a review of in-package chemistry modeling and a review of neutron-absorber material studies performed to date.

Long term exposure tests will also be performed on alternative neutron absorber materials (Table 2.2.14.2). This experimental matrix includes alternative borated SS materials, Ni-Gd and future materials such as rapidly solidified thermal spray materials.

Table 2.2.14.1. Task 14, FY11-17 Matrix 1: Long term exposure testing of 304-B SS (402 samples).

[Cl^-] / *m*	[NO_3^-] / *m*	Other composition[1]	T (ºC)	pH	# Samples of each material analyzed at each time period[2]								
					0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
___	___	TBD-1	30	___	6,0	6,0	6,0	6, 3	6,3	6,3	10,3	10,3	5,3
___	___	TBD-1	60	___	6,0	6,0	6,0	6, 3	6,3	6,3	10,3	10,3	5,3
___	___	TBD-1	90	___	6,0	6,0	6,0	6, 3	6,3	6,3	10,3	10,3	5,3
___	___	TBD-2	30	___	6,0	6,0	6,0	6, 3	5,3	5,3	10,3	10,3	5,3
___	___	TBD-2	60	___	6,0	6,0	6,0	6, 3	5,3	5,3	10,3	10,3	5,3
___	___	TBD-2	90	___	6,0	6,0	6,0	6, 3	5,3	5,3	10,3	10,3	5,3

[1] The environments will be determined from an analysis of in-package chemistry modeling and review of neutron-absorber material studies performed to date.

[2] The number of samples is given as *a*, *b* where *a* = # weight loss samples, *b* = # of crevice samples.

NOTE: All neutron absorber material testing will be performed in dedicated glassware or exposure tanks to avoid cross-contamination of boron from experiment to experiment.

Table 2.2.14.2. Task 14, FY11-17 Matrix 2: Long term exposure testing of alternative neutron absorber materials (360 samples).

Environment: TBD									
Material	# Samples analyzed at each time period, per environment, per material[1]								
	0.1 yr	0.25 yr	0.5 yr	1 yr	2 yr	5 yr	10 yr	20 yr	Extra
Alternative borated SS 1	5,0	5,0	5,0	5,3	5,3	5,3	5,3	5,3	5,3
Alternative borated SS 2	5,0	5,0	5,0	5,3	5,3	5,3	5,3	5,3	5,3
Ni-Gd	5,0	5,0	5,0	5,3	5,3	5,3	5,3	5,3	5,3
Future material	5,0	5,0	5,0	5,3	5,3	5,3	5,3	5,3	5,3

[1] The number of samples is given as *a*, *b* where *a* = # weight loss samples, *b* = # of crevice samples.

NOTE: All neutron absorber material testing will be performed in dedicated glassware or exposure tanks to avoid cross-contamination of boron from experiment to experiment.

2.2.15 Task 15: Analysis of Stainless Steel Corrosion Products

The objective of this task is to provide information to radionuclide transport analysis and in-package chemistry modeling on the composition of corrosion products from 304 SS and 316 SS.

Samples of Stainless Steel Type 316 that have been exposed to repository-relevant environments in the LTCTF will be analyzed for chemical composition and, if possible, structure. Imaging and surface analytical techniques will include SEM, SIMS and XPS, AES or EDS. It is anticipated that three samples from three environments (SAW, SCW, SDW) will be examined with approximately ten analysis per condition for a total of 90 tests, including imaging. These activities will be performed in FY10.

2.2.16 Task 16: Corrosion Testing Under Dripping Conditions

The objective of this task is to improve confidence that corrosion models developed using inundated testing do not underestimate likelihood or extent of processes under dripping conditions; provide knowledge of any enhancement in corrosion rate or likelihood of initiation that is attributable to testing under inundated conditions vs. dripping conditions; and, where appropriate, provide data to performance margin analyses.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for general and localized corrosion behavior of Alloy 22 and titanium alloys described, respectively, in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007 [DIRS180778]) and *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in these documents.

This task will require a testing capability (possibly multiple exposure systems) to be constructed that has the following attributes:

- Temperature control of test samples and test chamber
- Control over RH in test chamber
- Control of dripping rate
- Control of chemical environment
- Access to samples for inspection / removal
- Ability to include instrumented samples (e.g., electrochemical measurements on coplanar electrodes or DCPD on CT samples for SCC testing).

Once the exposure chamber(s) are constructed, an experimental program will be undertaken to generate data under dripping conditions that can be directly compared to Project data collected under inundated conditions. The specific corrosion modes that will be investigated are summarized in Table 2.2.16.1 and include: general corrosion rate, crevice corrosion and stress corrosion cracking.

Table 2.2.16.1. Task 16, FY11-17 Matrix 1: Rough order estimate of testing under dripping conditions.

Goal	Notes	Approximate number of Samples	Approximate Duration
Characterize weight-loss behavior of Alloy 22 and Titanium Grades 7 and 29	-	300 (2 environments[1], 3 materials, 5 time steps: 0.25, 0.5, 1, 2, 5 years, 10 samples per time step)	5 years
Characterize crevice initiation and propagation for Alloy 22, an Alloy 22 surrogate, and Titanium Grades 7 and 29	The Alloy 22 surrogate will be a material that has been observed to undergo crevice corrosion in a similar, inundated environment. This will lend insight into how extent of damage depends upon inundated vs. dripping.	120 (2 environments[1], 4 materials, 3 time steps: 1, 2 and 5 years, 5 samples per time step)	5 years
Characterize SCC initiation on Alloy 22 and Titanium Grades 7 and 29	Samples will be double U-bend configuration	94 (2 environments[1], 3 materials, 3 time steps: 1, 2, 5 years, 5 samples per time step)	5 years
Characterize SCC initiation and propagation on Alloy 22 and Titanium Grades 7 and 29	Samples will be static weight-loaded CT-type samples monitored with DCPD	18 (2 environments[1], 3 materials, continuous monitoring, 3 samples per configuration)	5 years
Characterize electrochemical behavior of Alloy 22, Alloy 22 surrogate, and Titanium Grades 7 and 29	Samples will be 2 or 3-electrode, coplanar configuration suitable for LPR or EIS experiments. Information will be sought on sample behavior and impedance of the adsorbed electrolyte layer.	24 (2 environments[1], 4 materials, continuous monitoring, 3 samples per configuration)	5 years
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	100	5 years

[1] One environment will contain chloride without nitrate and one environment will contain chloride and nitrate. Candidate environments include SCW, 1 m NaCl or electrolytes that will concentrate to these conditions (e.g., SDW, 0.1 m NaCl). If additional environments can be tested, a valid approach would be to test in SDW at low (e.g, 60ºC) temperature to simulate the cool period (e.g., temperature < boiling on waste package) and to test in SDW with heated samples such that evaporation occurs. A two cycle exposure could also be performed where a high temperature pre-conditioning enables salt deposits to form on the sample which would then be exposed to lower temperature conditions where low concentration electrolyte would contact the previously deposited salt layers.

2.2.17 Task 17: Open Circuit Potential of Titanium

The objectives of this task are to improve confidence in crevice corrosion initiation models and predictions; improve understanding of the influence of surface finish, physical environment (contact with tuff) and chemical environment on open circuit potential; and provide data to performance margin analysis activities.

Pre-test predictions for testing activities are outside the scope of this planning document and will be described in the TWP developed to direct specific activities. The criteria for defining the predictions for activities covered in this task will be based upon the models for localized corrosion behavior of titanium alloys described in *General Corrosion and Localized Corrosion of the Drip Shield* (SNL 2007 [DIRS 180778]). Analyses of data from this task will, in turn, improve upon the models and processes described in this document.

Long term testing will investigate the effects of contact with tuff, electrochemical environment and surface finish on the E_{CORR} value for Titanium Grade 7 and Titanium Grade 29 (Table 2.2.17.1).

Table 2.2.17.1. Task 17, FY11-17 Matrix 1: Open circuit monitoring of titanium alloys.

Goal	Notes	Approximate number of Samples[1]	Approximate Duration
Effect of contact with tuff on Ecorr of Titanium Grade 7 and Titanium Grade 29	Ecorr will be measured in the presence of crushed tuff. Baseline environments will include: initially DI H_2O, a moderate Cl^- environment (e.g., pH 6, 0.5 m) and a chloride + nitrate environment	42 (24, 18) (1 temperature, 3 environments, 2 materials, 4 echem samples per condition, 3 witness coupons per condition)	2 years
Effect of surface finish on Ecorr of Titanium Grade 7 and Ti Grade 29	Surface finishes include as-fabricated, polished, thermal oxide and deliquescent exposure.	80 (32, 48)[2] (1 temperature, 1 environment, 2 materials, 4 surface finishes, 4 echem samples per condition, 6 witness coupons per condition.	5 years(2)
TBD	As information becomes available from other test efforts, it is anticipated that additional testing will be performed to improve confidence and reduce uncertainties.	20	5 years

[1] The number of samples is given as *x (y, z)*; where *x* = the total number of samples, *y* = the number of electrochemically monitored samples (e.g., rods), and *z* = the number of witness samples (e.g., foils) to be removed at various time steps for archiving or surface analysis. Witness coupons may be removed dependent on analysis of the open circuit potential: that is, samples should be removed after the open circuit potential has apparently changed by a significant amount or after a prescribed length of time.

[2] It is anticipated that non-steady state surface finishes will evolve slowly toward a steady state structure and this process may require a significant exposure period. Thus, this activity has an increased number of witness samples and an increased duration compared to the other open circuit activities.

3. FACILITIES

This section of the document discusses:

- The selection process for assigning facility types to specific test activities (Section 3.1).
- The gap analysis comparing facility needs based on test activities to available facilities (Section 3.2).
- The additional facility requirements to meet testing needs not satisfied by currently available facilities (Section 3.3).
- Lessons learned from previous tests conducted at the LTCTF (Section 3.4).

3.1 Facility Selection

3.1.1 Process used to develop criteria for facility selection

A meeting was held between Lead Lab and DOE on October 16-17, 2007 to discuss the drivers that contribute to selection of a suitable test facility to meet a specific testing goal. In attendance were Paige Russell (DOE), Dennis Thomas (LL), Neil Brown (LL) and Doug Wall (LL).

The elements that were identified as necessary for a successful testing program were the following:

- Long term tests require a test facility that will be stable over the course of the testing. Facilities for tests in this category are essentially limited to DOE controlled institutions such as national laboratories.
- The test program should be adaptable to the extent possible; as the testing needs evolve different capabilities may be required.
- The test program should be responsive to changes in scope and budget; ideally the critical personnel and capabilities will be maintained through budgetary cycles. Accomplishing this goal may be possible through a combination of using private contractors for portions of the work and utilizing National Lab personnel who are not solely dependent on the Yucca Mountain Program (YMP) for support.
- The test program should have access to expertise and supporting technologies such as surface analytical equipment. The test facilities may vertically integrate specific key technologies where appropriate; however, the goal will be to leverage service organizations where possible and appropriate. This may require that some testing be physically located near the analysis capabilities.
- Project knowledge should be maintained by having at least a portion of the testing performed by key personnel who have continuity in the project and will be available in the future to support project requests.

3.1.2 Criteria for facility selection

Consideration of the elements outlined in Section 3.1 resulted in the establishment of two guiding criteria for what facilities should be selected for specific testing tasks.

1. Testing that is of long duration (e.g., multiple years) or developmental in nature (e.g., new techniques) should be performed at a national lab or other DOE controlled facility.

2. Testing that is of short duration (e.g.., less than two years) and follows a developed or established procedure may be performed either at a national lab, other DOE controlled facility, or by a private contractor including universities.

These criteria were applied to the test activities described in Section 2.2 and a matrix of facility recommendations was generated (see Section 3.3). Some of the stress corrosion cracking work is performed at GEGRC, which has a long track record in that arena and a long term relationship with the project.

3.1.3 Facility selection for testing activities

The test matrices in Section 2.2 of this document are summarized in Table 3.1.3.1 along with recommendations for the facility type where each activity should be carried out. The criteria used to determine the appropriate facility type are found in Section 3.1.2. There are two exceptions where the criteria are modified as described in the following paragraphs.

Some activities require performing many short duration tests to complete the proposed matrix of conditions: each test is short but the entire test matrix may take 2 or more years. In this case, it would be straightforward to shut down and move the testing activity if necessary. Thus activities that are highly portable and follow an established procedure, even though long in duration, may be performed by a private contractor.

The second exception pertains to the work planned to be performed at GE Global R&D Center. GEGRC has provided high quality data and planning assistance to the Project for a number of years. The test facility has much capability that is not duplicated elsewhere on the project and the personnel include world-class experts in the areas of environmental cracking and corrosion science. GEGRC is a continuing performer of long duration experiments and also contributes to the development of testing approaches. Thus future activities identified with GEGRC will continue to include development and long term testing activities and this is reflected in Table 3.1.3.1.

In Table 3.1.3.1 under the *Recommended Facility Type* heading, the designation of National Lab also includes any other appropriate DOE controlled facilities.

Table 3.1.3.1. Facility selection by Task, Fiscal Year and Matrix Number.

Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Recommended Facility Type
1	08	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	National Lab or contractor
1	09	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	National Lab or contractor
1	10	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	National Lab or contractor
1	11+	1	Rough order estimate of long-term weight-loss coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29	20	Long-term aqueous exposures, Weight loss, Microscopy	National Lab
1	11+	2	Rough order estimate of long-term weight-loss coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials	20	Long-term aqueous exposures, Weight loss, Microscopy	National Lab
2	08	1	Crevice specimens to be photographed and examined for signs of localized corrosion attack	1	Microscopy	National Lab or contractor
2	09	1	Prioritization of Crevice Samples for Detailed Analysis	1	Microscopy	National Lab or contractor
2	09	2	Detailed Analysis of Priority 1 Samples from Task 2, FY09, Matrix 1	1	Microscopy	National Lab or contractor
2	10	1	Detailed Analysis of Priority 2,3 and 4 Samples from Task 2, FY09, Matrix 1	1	Microscopy	National Lab or contractor
2	10	2	Scoping Experiments to Characterize Alloy 22 Crevice Corrosion at Open Circuit Potential.	1	Aqueous exposures, Microscopy	National Lab or contractor
2	11+	1	Rough order estimate of long-term crevice coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29	20	Long-term aqueous exposures, Microscopy	National Lab or contractor
2	11+	2	Rough order estimate of long-term crevice coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials	20	Long-term aqueous exposures, Microscopy	National Lab or contractor
2	11+	3	Rough order estimate of crevice corrosion initiation and propagation testing under open circuit conditions for Alloy 22	5	Long-term aqueous exposures, Microscopy	National Lab or contractor
3	08	1	Electrochemical testing of Alloy 22 crevice corrosion behavior to reproduce critical potential data that were excluded from the localized corrosion model	1	Bench top electrochemical tests, microscopy	National Lab or contractor
3	09	1	Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0.1 m NO_3^- with temperatures between 20°C and 95°C.	1	Bench top electrochemical tests, microscopy	National Lab or contractor
3	10	1	Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0 m NO_3^- with temperatures between 20°C and 95°C.	1	Bench top electrochemical tests, microscopy	National Lab or contractor
3	11+	1	Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of Alloy 22	1-2	Bench top electrochemical tests, microscopy	National Lab or contractor
4	08	1	Experiments to reproduce data from conditions where a large degree of variability was observed in the open circuit response.	1-2	Aqueous exposures, open circuit measurements Microscopy	National Lab
4	09-11	1	Systematic Study of the Effect of Environmental Variables On the Open Circuit Potential of Alloy 22	3	Aqueous exposures, open circuit measurements Microscopy	National Lab
4	10	1	Open Circuit Experiments to Determine the Effect of Pre-exposure to Deliquescent Environments	1-2	Aqueous exposures, open circuit measurements Microscopy	National Lab

Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Recommended Facility Type
4	11+	1	Rough order estimate of open circuit testing on Alloy 22 to determine the influence of secondary environmental parameters	2-5	Aqueous exposures, open circuit measurements Microscopy	National Lab
5	08	1	Electrochemical testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	National Lab
5	08	2	Weight loss testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures	National Lab
5	09	1	Weight loss testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1	Microscopy, weight loss	National Lab or contractor
5	09	2	Weight loss testing to measure the temperature dependence of titanium alloys in seepage type electrolytes	1-2	Aqueous exposures	National Lab
5	09	3	Electrochemical testing to measure Titanium Grade 29 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	National Lab
5	09	4	Short-term weight loss testing (12 weeks) of Alloy 22 and Titanium materials at 120°C, 150ºC and 180ºC to measure temperature dependence in deliquescent brine type electrolytes	1	Aqueous exposures	National Lab or contractor
5	10	1	Weight loss testing to measure the temperature dependence of titanium alloys in seepage type electrolytes	1	Microscopy, weight loss	National Lab or contractor
5	10	2	Electrochemical testing to measure Titanium Grade 7 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	National Lab
5	10	3	Weight loss testing of Alloy 22 and Titanium materials exposed to steam and humid environments	1-2	Atmospheric chamber exposures, microscopy, weight loss	National Lab
5	10	4	Short-term weight loss testing (12 weeks) of Alloy 22 and Titanium materials at 120°C, 150ºC and 180ºC to measure temperature dependence in deliquescent brine type electrolytes	1	Microscopy, weight loss	National Lab or contractor
5	11+	1	Electrochemical testing to measure Titanium Grade 28 (or grade 7 / grade 28/grade 29 welds) temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	National Lab
5	11+	2	Medium duration testing of Alloy 22 in deliquescent brine chemistries	1-2	Aqueous exposures, microscopy, weight loss	National Lab
5	11+	3	Electrochemical measurement of corrosion rate in deliquescent brine chemistries	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	National Lab
6	08	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 7	1	Bench top electrochemical tests, microscopy	National Lab or contractor
6	09	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 29	1	Bench top electrochemical tests, microscopy	National Lab or contractor
6	10	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 28	1	Bench top electrochemical tests, microscopy	National Lab or contractor
6	11+	1	Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of titanium alloys and welds	1	Bench top electrochemical tests, microscopy	National Lab or contractor

Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Recommended Facility Type
7	08	1	Characterization of Alloy 22 oxides on specimens exposed for 5 years in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	National Lab
7	09	1	Characterization of Alloy 22 oxides on specimens exposed for 9.5 years in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	National Lab
7	10	1	Characterization of Alloy 22 on specimens exposed to deliquescent conditions	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	National Lab
7	11+	1	Summary of oxide characterization activities in FY11-17	1-5	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM), bench top / specialized electrochemical tests	National Lab
7	11+	2	Analysis of Alloy 22 oxides from samples exposed to aqueous solutions following deliquescent pre-exposure	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	National Lab
8	08	1	Evaluation of sample configurations and monitoring techniques for corrosion studies under deliquescent conditions	1	Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests	National Lab
8	08	2	Corrosion behavior under deliquescent conditions of Alloy 22 and less-corrosion resistant materials as a function of mass-loading, temperature and dewpoint	1	Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests	National Lab
8	08	3	Evaluation of the stability of salt and salt assemblages in high-temperature, high dewpoint environments	1	Deliquescent exposures, microscopy, XRD	National Lab
8	09	1	Effect of Deliquescent Conditions on Oxide Stability (provides comparison to weight loss studies from Task 5 and supplies samples to oxide characterization studies in Task 7)	1	Deliquescent exposures, solid contamination, microscopy	National Lab
8	10	1	Effect of Deliquescent Conditions on Open Circuit Behavior in Seepage Environments (provides input to Tasks 4 and 7)	1	Deliquescent exposures, solid contamination, microscopy	National Lab
8	11+	1	Summary of long-term testing under deliquescent environments	5	Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests	National Lab
9	08	1	Analysis of MIC samples from testing in growth media	1	microscopy, FIB, image analysis	National Lab
9	11+	1	Rough order estimate of out year MIC testing to be performed on Alloy 22	5	exposure to microbe consortia, controlled RH,temperatureand contaminants, microscopy	National Lab

Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Recommended Facility Type
10	08	1	Comparison of aged Alloy 22 compositions with the current model range and the ASTM range	NA	NA (matrix for information only)	NA (matrix for information only)
10	08	2	Summary of calculations and sample characterization to be performed in FY08	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	National Lab or contractor
10	09	1	Analysis of Ni-Cr-Mo samples aged for 105 hours	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	National Lab or contractor
10	10	1	Analysis of Alloy 22 samples aged for 104 hours	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	National Lab or contractor
10	11+	1	Summary of out year activities for aging and phase stability of Alloy 22	5	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	National Lab
11	08	1	Constant load Keno tests in aerated, 105ºC, 15% BSW seepage type brine	~4	Multiple specimen constant load gas purged autoclave	National Lab or Contractor (GEGRC)
11	08	2	Creep testing of titanium Grades 7, 28 and 29	1	Constant load test	National Lab or Contractor (GEGRC)
11	08	3	Single U-bend testing in 165ºC aerated SCW brine	<10	Gas purged autoclave	National Lab or Contractor (GEGRC)
11	08	4	Double U-Bend testing in 165ºC aerated SCW brine	<10	Gas purged autoclave	National Lab or Contractor (GEGRC)
11	08	5	Weld defect tolerance specimens in 105ºC, 15% BSW seepage type brine	4	Gas purged autoclave, reverse DC potential drop	National Lab or Contractor (GEGRC)
11	08	6	Electrolyte compositions for electrochemical testing of Alloy 22 and titanium alloys	NA	General chemistry	National Lab or Contractor (GEGRC)
11	09/ 10	1	Constant load testing	4	Constant load testing	National Lab or Contractor (GEGRC)
11	09/ 10	2	U-Bend tests	<10	Gas purged autoclave	National Lab or Contractor (GEGRC)
11	09/ 10	3	Double U-Bend tests	<10	Gas purged autoclave	National Lab or Contractor (GEGRC)
11	09/ 10	4	Weld defect tolerance specimens	4	Constatnt load gas purged autoclave	National Lab or Contractor (GEGRC)
11	09/ 10	5	Test environments for long-term electrochemical measurements	NA	General chemistry	National Lab or Contractor (GEGRC)
11	11+	1	Rough order estimate of long-term double U-bend sample testing for Alloy 22, and Titanium Grades 7 and 29	20	Long term aqueous exposures, microscopy	National Lab
11	11+	2	Rough order estimate of long-term double U-bend sample testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials	20	Long term aqueous exposures, microscopy	National Lab

Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Recommended Facility Type
11	11+	3	Rough order estimate of long-term CT-sample testing for Alloy 22, Alloy 22 surrogates, Titanium Grade 7 and Titanium Grade 29	5+	Long term aqueous exposures, microscopy, controlled load apparatus, crack length monitoring	National Lab or contractor (GEGRC)
12	08	1	Examination of select 9.5 year Alloy 22 U-bend specimens	1	microscopy	National Lab or contractor
12	08	2	Examination of select 6.6 year Titanium Grade 7 U-bend specimens	1	microscopy	National Lab or contractor
12	08	3	Examination of select 9.5 year Titanium Grade 16 U-bend specimens	1	microscopy	National Lab or contractor
12	09	1	Examination of 9.5 year Alloy 22 U-bend specimens	1	microscopy	National Lab or contractor
12	09	1	Examination of 6.6 year Titanium Grade 7 U-bend specimens	1	microscopy	National Lab or contractor
12	09	1	Examination of 9.5 year Titanium Grade 16 U bend specimens	1	microscopy	National Lab or contractor
13	10	1	Establish Relationship between Bulk Hydrogen Content and Mechanical Properties for Titanium Grade 7 and Alloy 22	1	mechanical testing, hydrogen charging, bulk hydrogen analysis	National Lab or contractor
13	11+	1	Rough Order Estimate for Testing to Support Hydrogen Embrittlement Studies	20	aqueous exposures, bench top electrochemical tests, thermal desorption spectroscopy, mechanical testing	National Lab
14	11+	1	Long term exposure testing of 304-B SS	20	Aqueous exposures, weight loss, microscopy	National Lab
14	11+	2	Long term exposure testing of alternative neutron absorber materials	20	Aqueous exposures, weight loss, microscopy	National Lab
15	10	NA	This task has a single activity for the analysis of corrosion products from 316 stainless steel exposed in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	National lab or contractor
16	11+	1	Rough order estimate of testing under dripping conditions	5	chamber for performing tests under dripping conditions, bench top electrochemical tests, weight loss, microscopy	National Lab
17	11+	1	Open circuit monitoring of titanium alloys	2-5	aqueous exposures, open circuit measurements, microscopy, weight loss	National Lab

3.2 Gap analysis

3.2.1 Summary of existing facilities and capabilities

The Project currently has access to infrastructure for corrosion testing that has been established over the past 10 plus years of experimental work. The primary facility for testing has been the LTCTF located at LLNL. In August of 2006 the LTCTF was decommissioned; however, some capability was retained. A significant portion of the LTCTF resources were in the form of long-term exposure tanks with a capacity of 2000 L per tank. Most of these tanks are no longer useable; though a small number of tanks (4) could be brought on-line to support future testing needs. The LTCTF also included an electrochemical test laboratory. Many of the capabilities of this laboratory (specimen preparation, high temperature brine exposures, long-term open circuit measurements, and potentiostat (60 units) controlled measurements) have been relocated to a new laboratory and are available to support project needs. A thermal aging facility comprised of 11 furnaces is also available for evaluation of alloy phase stability. Several exposure ovens are available for generating exposure environments for deliquescence testing, although only 100% water saturation can currently be used for Q measurements.

A significant component of the SCC work on alloy 22 and titanium has been performed under contract at GEGRC in Schenectady, New York. Instrumented, feedback-controlled load frames are used to perform controlled stress intensity experiments and measure crack growth kinetics on compact tension specimens. A novel test apparatus has also been assembled for testing many tensile bar samples at constant load simultaneously (i.e., the Keno test). It is anticipated that these resources will be available over the next several years to perform Project testing although the long-term availability is unknown.

A new facility at Sandia National Laboratories (SNL) has been established for testing under deliquescent conditions. A chamber capable of testing at over 200ºC at ambient pressure with controlled air/moisture levels has been brought on-line. This facility is dedicated to future testing for the Yucca Mountain Project. Additional laboratory space is also available at SNL to support bench-top type electrochemical and exposure testing. A dedicated cleaning laboratory and analytical support department are also available for prepping, cleaning and examining corrosion specimens, including oxide layer characterization.

Additional resources for electrochemical testing and specimen characterization are available to the corrosion test program through national labs (e.g., Idaho National Labs (INL)) and contract organizations (e.g., Corrosion Test Laboratories (CTL)).

3.2.2 Identification of gap between testing needs and available facilities

The information compiled in Table 3.1.3.1 has been used to assess the ability of currently available facilities (Section 3.2.1) to accommodate the testing needs from FY08 through FY17. A gap analysis is presented in Table 3.2.2.1 that lists each test activity and identifies whether or not the Project currently has access to facilities that can meet the testing needs. The activities that require additional facility capabilities are shown in bold with shading.

The primary activities that require additional facilities are the long term exposures (up to 20 years) of weight loss specimens, crevice specimens and SCC specimens (see Table 3.2.2.1, rows 4, 5, 11, 12, 68, 69, 70, 79 and 80). These activities will require exposure tanks with capacities ranging from 100 to 200 gallons. The tanks will need to be maintained for up to at least 20 years. The laboratory space to accommodate this testing is currently not available to the Project.

Long term open circuit potential measurements (Table 3.2.2.1, row 13) could potentially be accommodated by current facilities; however, these tests may continue for a number of years and the currently available facilities are not intended for this length of exposure testing. For this reason this activity is categorized as not supported by current facility capabilities. This activity will require electrochemically monitored exposure systems (bench-top) to be maintained for 3-5 years.

Long term deliquescent environment exposures (up to 5 years) cannot be accommodated by the currently available facilities (Table 3.2.2.1, row 49). Although a deliquescent exposure system exists at SNL, this facility is not intended for long term exposures and does not have the capacity to accommodate a large sample set. Furthermore, it is likely that more than one additional exposure system will be required to simultaneously test different exposure conditions.

A facility currently does not exist to assess the long term effects of exposing metals to environments favorable to MIC (Table 3.2.2.1, row 51). The design and procurement of an appropriate exposure chamber is included in the Task 9 activities planned for FY09 and FY10.

The hydrogen embrittlement studies planned for FY11 through FY17 cannot be accommodated by currently available facilities (Table 3.2.2.1, row 78). The hydrogen embrittlement studies require the fabrication of a thermal desorption spectroscopy (TDS) system. Additionally, long term sample exposures will feed some of the hydrogen embrittlement activities. These exposures will be accommodated by the facilities and test equipment used to support the long term sample exposures for weight loss, crevice and SCC specimens.

A facility for testing under simulated dripping conditions does not currently exist (Table 3.2.2.1, Row 82). This capability development is part of the FY11-17 activities for Task 16. The laboratory space to accommodate such a capability is not currently available to the Project and will need to be included in a future test facility.

All of the activities described above as requiring facilities not currently available are planned for FY11 or later. The balance of the experimental work, e.g., all activities planned for FY08 through FY10 and much of the FY11+ work, can be accommodated by the facilities currently available to the Project. These facilities include National Labs and private contractors as described in Section 3.2.1.

Based on this gap analysis, a recommendation for facility capabilities and infrastructure requirements to support FY11+ testing is developed in the following section of this document (Section 3.2.3.).

Table 3.2.2.1. Gap analysis between existing facility resources and testing needs

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
1	1	08	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	Y
2	1	09	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	Y
3	1	10	1	Weight loss and crevice specimens to be photographed and weighed	1	Microscopy, Weight Loss	Y
4	**1**	**11+**	**1**	**Rough order estimate of long-term weight-loss coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29**	**20**	**Long-term aqueous exposures, Weight loss, Microscopy**	**N**
5	**1**	**11+**	**2**	**Rough order estimate of long-term weight-loss coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials**	**20**	**Long-term aqueous exposures, Weight loss, Microscopy**	**N**
6	2	08	1	Crevice specimens to be photographed and examined for signs of localized corrosion attack	1	Microscopy	Y
7	2	09	1	Prioritization of Crevice Samples for Detailed Analysis	1	Microscopy	Y
8	2	09	2	Detailed Analysis of Priority 1 Samples from Task 2, FY09, Matrix 1	1	Microscopy	Y
9	2	10	1	Detailed Analysis of Priority 2,3 and 4 Samples from Task 2, FY09, Matrix 1	1	Microscopy	Y
10	2	10	2	Scoping Experiments to Characterize Alloy 22 Crevice Corrosion at Open Circuit Potential.	1	Aqueous exposures, Microscopy	Y
11	**2**	**11+**	**1**	**Rough order estimate of long-term crevice coupon testing for Alloy 22, Titanium Grade 7 and Titanium Grade 29**	**20**	**Long-term aqueous exposures, Microscopy**	**N**
12	**2**	**11+**	**2**	**Rough order estimate of long-term crevice coupon testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials**	**20**	**Long-term aqueous exposures, Microscopy**	**N**
13	**2**	**11+**	**3**	**Rough order estimate of crevice corrosion initiation and propagation testing under open circuit conditions for Alloy 22**	**5**	**Long-term aqueous exposures, Microscopy**	**N**
14	3	08	1	Electrochemical testing of Alloy 22 crevice corrosion behavior to reproduce critical potential data that were excluded from the localized corrosion model	1	Bench top electrochemical tests, microscopy	Y
15	3	09	1	Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0.1 m NO_3^- with temperatures between 20°C and 95°C.	1	Bench top electrochemical tests, microscopy	Y
16	3	10	1	Determine threshold values for Cl^- concentration at which Alloy 22 is not susceptible to crevice corrosion initiation in 0 m NO_3^- with temperatures between 20°C and 95°C.	1	Bench top electrochemical tests, microscopy	Y

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
17	3	11+	1	Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of Alloy 22	1-2	Bench top electrochemical tests, microscopy	Y
18	4	08	1	Experiments to reproduce data from conditions where a large degree of variability was observed in the open circuit response.	1-2	Aqueous exposures, open circuit measurements Microscopy	Y
19	4	09-11	1	Systematic Study of the Effect of Environmental Variables On the Open Circuit Potential of Alloy 22	3	Aqueous exposures, open circuit measurements Microscopy	Y
20	4	10	1	Open Circuit Experiments to Determine the Effect of Pre-exposure to Deliquescent Environments	1-2	Aqueous exposures, open circuit measurements Microscopy	Y
21	4	11+	1	Rough order estimate of open circuit testing on Alloy 22 to determine the influence of secondary environmental parameters	2-5	Aqueous exposures, open circuit measurements Microscopy	Y
22	5	08	1	Electrochemical testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	Y
23	5	08	2	Weight loss testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures	Y
24	5	09	1	Weight loss testing to measure Alloy 22 temperature dependence in seepage type electrolytes	1	Microscopy, weight loss	Y
25	5	09	2	Weight loss testing to measure the temperature dependence of titanium alloys in seepage type electrolytes	1-2	Aqueous exposures	Y
26	5	09	3	Electrochemical testing to measure Titanium Grade 29 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	Y
27	5	09	4	Short-term weight loss testing (12 weeks) of Alloy 22 and Titanium materials at 120°C, 150ºC and 180ºC to measure temperature dependence in deliquescent brine type electrolytes	1	Aqueous exposures	Y
28	5	10	1	Weight loss testing to measure the temperature dependence of titanium alloys in seepage type electrolytes	1	Microscopy, weight loss	Y
29	5	10	2	Electrochemical testing to measure Titanium Grade 7 temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	Y
30	5	10	3	Weight loss testing of Alloy 22 and Titanium materials exposed to steam and humid environments	1-2	Atmospheric chamber exposures, microscopy, weight loss	Y

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
31	5	10	4	Short-term weight loss testing (12 weeks) of Alloy 22 and Titanium materials at 120°C, 150ºC and 180ºC to measure temperature dependence in deliquescent brine type electrolytes	1	Microscopy, weight loss	Y
32	5	11+	1	Electrochemical testing to measure Titanium Grade 28 (or grade 7 / grade 28/grade 29 welds) temperature dependence in seepage type electrolytes	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	Y
33	5	11+	2	Medium duration testing of Alloy 22 in deliquescent brine chemistries	1-2	Aqueous exposures, microscopy, weight loss	Y
34	5	11+	3	Electrochemical measurement of corrosion rate in deliquescent brine chemistries	1-2	Aqueous exposures, bench top electrochemical tests, microscopy	Y
35	6	08	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 7	1	Bench top electrochemical tests, microscopy	Y
36	6	09	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 29	1	Bench top electrochemical tests, microscopy	Y
37	6	10	1	Testing to determine the combined effects of chloride, nitrate and fluoride on critical potential of Titanium Grade 28	1	Bench top electrochemical tests, microscopy	Y
38	6	11+	1	Rough order estimate of electrochemical testing to support evaluating crevice corrosion behavior of titanium alloys and welds	1	Bench top electrochemical tests, microscopy	Y
39	7	08	1	Characterization of Alloy 22 oxides on specimens exposed for 5 years in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	Y
40	7	09	1	Characterization of Alloy 22 oxides on specimens exposed for 9.5 years in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	Y
41	7	10	1	Characterization of Alloy 22 on specimens exposed to deliquescent conditions	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	Y
42	7	11+	1	Summary of oxide characterization activities in FY11-17	1-5	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM), bench top / specialized electrochemical tests	Y

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
43	7	11+	2	Analysis of Alloy 22 oxides from samples exposed to aqueous solutions following deliquescent pre-exposure	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	Y
44	8	08	1	Evaluation of sample configurations and monitoring techniques for corrosion studies under deliquescent conditions	1	Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests	Y
45	8	08	2	Corrosion behavior under deliquescent conditions of Alloy 22 and less-corrosion resistant materials as a function of mass-loading, temperature and dewpoint	1	Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests	Y
46	8	08	3	Evaluation of the stability of salt and salt assemblages in high-temperature, high dewpoint environments	1	Deliquescent exposures, microscopy, XRD	Y
47	8	09	1	Effect of Deliquescent Conditions on Oxide Stability (provides comparison to weight loss studies from Task 5 and supplies samples to oxide characterization studies in Task 7)	1	Deliquescent exposures, solid contamination, microscopy	Y
48	8	10	1	Effect of Deliquescent Conditions on Open Circuit Behavior in Seepage Environments (provides input to Tasks 4 and 7)	1	Deliquescent exposures, solid contamination, microscopy	Y
49	**8**	**11+**	**1**	**Summary of long-term testing under deliquescent environments**	**5**	**Deliquescent exposures, solid contamination, DCPD, microscopy, weight loss, bench top electrochemical tests**	**N**
50	9	08	1	Analysis of MIC samples from testing in growth media	1	microscopy, FIB, image analysis	Y
51	**9**	**11+**	**1**	**Rough order estimate of out year MIC testing to be performed on Alloy 22**	**5**	**exposure to microbe consortia, controlled RH,temperaturea nd contaminants, microscopy**	**N**
52	10	08	1	Comparison of aged Alloy 22 compositions with the current model range and the ASTM range	NA	NA (matrix for information only)	NA
53	10	08	2	Summary of calculations and sample characterization to be performed in FY08	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	Y

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
54	10	09	1	Analysis of Ni-Cr-Mo samples aged for 105 hours	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	Y
55	10	10	1	Analysis of Alloy 22 samples aged for 104 hours	1	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	Y
56	10	11+	1	Summary of out year activities for aging and phase stability of Alloy 22	5	microscopy, EBSD, phase volume fraction calculation, Calphad-ThermoCalc	Y
57	11	08	1	Constant load Keno tests in aerated, 105ºC, 15% BSW seepage type brine	~4	Multiple specimen constant load gas purged autoclave	Y
58	11	08	2	Creep testing of titanium Grades 7, 28 and 29	1	Constant load test	Y
59	11	08	3	Single U-bend testing in 165ºC aerated SCW brine	<10	Gas purged autoclave	Y
60	11	08	4	Double U-Bend testing in 165ºC aerated SCW brine	<10	Gas purged autoclave	Y
61	11	08	5	Weld defect tolerance specimens in 105ºC, 15% BSW seepage type brine	4	Gas purged autoclave, reverse DC potential drop	Y
62	11	08	6	Electrolyte compositions for electrochemical testing of Alloy 22 and titanium alloys	NA	General chemistry	Y
63	11	09/ 10	1	Constant load testing	4	Constant load testing	Y
64	11	09/ 10	2	U-Bend tests	<10	Gas purged autoclave	Y
65	11	09/ 10	3	Double U-Bend tests	<10	Gas purged autoclave	Y
66	11	09/ 10	4	Weld defect tolerance specimens	4	Constatnt load gas purged autoclave	Y
67	11	09/ 10	5	Test environments for long-term electrochemical measurements	NA	General chemistry	Y
68	**11**	**11+**	**1**	**Rough order estimate of long-term double U-bend sample testing for Alloy 22, and Titanium Grades 7 and 29**	**20**	**Long term aqueous exposures, microscopy**	**N**
69	**11**	**11+**	**2**	**Rough order estimate of long-term double U-bend sample testing for thermally or mechanically modified barrier materials, galvanic couples and surrogate materials**	**20**	**Long term aqueous exposures, microscopy**	**N**
70	**11**	**11+**	**3**	**Rough order estimate of long-term CT-sample testing for Alloy 22, Alloy 22 surrogates, Titanium Grade 7 and Titanium Grade 29**	**5+**	**Long term aqueous exposures, microscopy, controlled load apparatus, crack length monitoring**	**N**
71	12	08	1	Examination of select 9.5 year Alloy 22 U-bend specimens	1	microscopy	Y

Row #	Task	FY	Matrix #	Title / Description	Duration (years)	Capability Type	Available facilities meet testing needs (Y/N)
72	12	08	2	Examination of select 6.6 year Titanium Grade 7 U-bend specimens	1	microscopy	Y
73	12	08	3	Examination of select 9.5 year Titanium Grade 16 U-bend specimens	1	microscopy	Y
74	12	09	1	Examination of 9.5 year Alloy 22 U-bend specimens	1	microscopy	Y
75	12	09	1	Examination of 6.6 year Titanium Grade 7 U-bend specimens	1	microscopy	Y
76	12	09	1	Examination of 9.5 year Titanium Grade 16 U bend specimens	1	microscopy	Y
77	13	10	1	Establish Relationship between Bulk Hydrogen Content and Mechanical Properties for Titanium Grade 7 and Alloy 22	1	mechanical testing, hydrogen charging, bulk hydrogen analysis	Y
78	**13**	**11+**	**1**	**Rough Order Estimate for Testing to Support Hydrogen Embrittlement Studies**	**20**	**aqueous exposures, bench top electrochemical tests, thermal desorption spectroscopy, mechanical testing**	**N**
79	**14**	**11+**	**1**	**Long term exposure testing of 304-B SS**	**20**	**Aqueous exposures, weight loss, microscopy**	**N**
80	**14**	**11+**	**2**	**Long term exposure testing of alternative neutron absorber materials**	**20**	**Aqueous exposures, weight loss, microscopy**	**N**
81	15	10	NA	This task has a single activity for the analysis of corrosion products from 316 stainless steel exposed in the LTCTF	1	Microscopy, surface analytical techniques (e.g., FIB, SEM, TEM, XPS, AES, EDS, AFM)	Y
82	**16**	**11+**	**1**	**Rough order estimate of testing under dripping conditions**	**5**	**chamber for performing tests under dripping conditions, bench top electrochemical tests, weight loss, microscopy**	**N**
83	17	11+	1	Open circuit monitoring of titanium alloys	2-5	aqueous exposures, open circuit measurements, microscopy, weight loss	Y

3.3 Additional Facility Capability Requirements

3.3.1 Facility requirements definition

Based on the gap analysis performed in Section 3.2, sufficient facilities are available to perform the testing activities planned for FY08 through FY10, while additional facilities will be required to support the testing activities planned for FY11 and beyond. The primary technical capability requirements that define the additional facility capabilities are the following:

- Long term immersion testing: up to 20 year exposures, approximately 40 environments, on the order of 25,000 samples.
- Long term open circuit measurements
- Long term exposures to deliquescent conditions
- Exposures to environments favorable to MIC
- Hydrogen embrittlement studies
- Exposure to simulated dripping conditions

A facility that incorporates the capabilities identified in the above list that also consolidates the existing capabilities could be accommodated in a single location with approximately 10,000 square feet of total facility space (such that all testing in FY11 and beyond would be supported by a single facility). The breakdown of the space requirements is given in Table 3.3.1.1. Decisions on how much of the FY11+ work to consolidate into one location will be made at a later date. The square footage estimate is a rough order of magnitude estimate based upon the test matrices described herein.

Table 3.3.1.1. Space requirements for new corrosion test facility.

Space designation	Required floor space (square feet)
Office space	2300
Sample storage	1800
Long term immersion testing (i.e., exposure tanks)	2500
Other testing activities (e.g., bench top electrochemical tests, long term open circuit testing, MIC, deliquescent environments, dripping environments, microscopy lab, chemical preparation, etc.)	3400
Total	10000 +/-2000

3.3.2 Schedule for obtaining additional capability

In order to accomplish the planned testing for FY11 and beyond, the new capabilities need to be available by FY11. A rough estimate schedule for meeting that goal is as follows:

- Fiscal year 2009 – Finalize additional facility infrastructure needs and location.
- Fiscal year 2009 – Start facility build out.
- Fiscal year 2010 – Purchase equipment and samples to support facility startup.
- Fiscal year 2011 – Execute facility startup.

3.4 Lessons Learned

Lessons learned from the operation of the LTCTF at LLNL provide good guidance for initiating planning of a next generation test facility. Due to changes in the testing scope and testing objectives, not all of the operations at the LTCTF are relevant to future testing; however, it is prudent to build upon past experience.

Corrosion of highly corrosion resistant materials such as Alloy 22 and titanium in relatively benign environments is extremely difficult to quantify due to limitations in measurement techniques and artifacts introduced from sample handling and cleaning. In order to overcome the issues that arise with detecting low corrosion rates, the LTCTF allowed very long-duration experiments to be performed, thus increasing the amount of corrosion and improving the signal-to-noise ratio for corrosion rate measurements. Testing at the LTCTF was initially intended to provide information to enable screening of candidate materials for use in the EBS. Both general and localized corrosion modes were tested by employing a variety of sample types and configurations:

- Weight-loss coupons
- Crevice assemblies
- U-bend samples
- Galvanic assemblies.

Environments for testing were maintained by filling half the volume of 1,000-liter tanks with test electrolyte. Samples were either tested directly in the electrolyte, at the waterline (half-in, half-out) or in the gas phase above the electrolyte. The solution level was maintained through appropriate additions of distilled waters and bath chemistry was periodically sampled and analyzed. Upon removal from the tanks, samples were examined optically and in some cases using SEM and other surface analytical techniques. Crevice and U-bend samples were examined for signs of SCC and crevice corrosion, respectively. Weight-loss and crevice samples were cleaned and weighed, and weight change was used to estimate corrosion rate over the exposure period.

Some of the lessons learned from using this facility, its experimental approach and observations resulting from resolution of Condition Report 11851 are listed below:

- A formal data quality objective process should be applied to document desired precision and accuracy to arrive at sample configurations, sample quantities, and equipment requirements to meet these objectives.

- Control samples were not a formal component of the long-term testing approach. Several representative blank coupons should be archived in a benign environment both in the cleaned and as-received condition.

- Sample cleaning should be conducted in a manner to demonstrate that additional cleaning would not result in higher estimated corrosion rates. All recommended

practices within ASTM standard G1-90 should be considered. Vibratory etching rather than stamping of samples should be considered to minimize contamination.

- Samples were not sufficiently cleaned prior to initial weighing and introduction into the exposure vessels. Prior to exposure, samples should be cleaned in a manner similar to final cleaning to remove artifacts associated with stamping, machining and other processing and to ensure that a consistent starting condition exists for all samples.

- Samples were not sufficiently characterized prior to testing, with the result that identifying differences from initial to final condition was not straightforward. Future sample weighing will be carried out to a higher level of precision, with measured blanks maintained to demonstrate accuracy of future weight measurements.

- Samples were not included in the long term exposures with an appropriate surface finish for straightforward surface analysis. Polished surfaces were typically 600 grit to represent the anticipated surface finish of the waste pacakges; additional samples with less surface roughness would enable more robust oxide characterization, including oxide thickness measurements.

- Samples with to-be-determined sample timeframes were not added to allow for flexibility with regards to extending the testing duration, obtaining intermediate results, or obtaining additional samples for improved sample statistics.

- Sample geometry was not appropriate for weight-loss measurements on a passive material: surface area-to-volume ratio was not maximized, thus limiting the sensitivity to weight change.

- Alloy 22 crevice geometry samples were not polished on both sides adding uncertainty as to the actual corrosion rate of the base metal due to artifacts caused by weight loss from the unpolished surface.

- Samples should be thoroughly rinsed and dried immediately after removal to minimize drying of salts on the surface which can result in optical artifacts.

- Samples were loaded so that corrosion products from one could fall onto another, possibly introducing different local environments for samples arranged in a vertical array.

- Half of the samples were tested in the vapor phase above the aqueous phase. The temperature of this phase was not precisely controlled; instead the aqueous phase temperature was maintained. The warm, humid vapor phase environment condensed on the underside of the test chamber covers and the condensate could drip onto the suspended vapor phase samples. Thus control o the environment for the vapor phase samples could be more precisely controlled by using a dedicated controlled humidity system with appropriate contaminates loaded onto the samples prior to exposure.

- Dissimilar metals were mixed in the same test vessel. This practice can introduce artifacts or unknowns into the analysis.
- There was no continuous electrochemical monitoring. The opportunity to significantly increase the data set and improve understanding of long-term behavior was lost. Electrochemical measurements are more sensitive to small corrosion changes than weight loss.
- The use of large testing tanks reduced the number of environments that could be tested and reduced the flexibility of the overall test program.
- The initial test program had such a large scope that adequate resources were not available at test completion to carry out a comprehensive analysis effort.
- The evolution of the test environment was not anticipated for samples tested in autoclaves at high temperature and pressure. The aggressiveness of the environments might have increased during the course of the test in such a way that the test conditions were not representative of environments that are stable at repository pressure.

4. REFERENCES

BSC (Bechtel SAIC Company) 2004, *Aging and Phase Stability of Waste Package Outer Barrier* [DIRS 171924], ANL-EBS-MD-000002 rev 02. Las Vegas, Nevada: Bechtel SAIC Company. ACC: DOC.20041005.0003.

BSC (Bechtel SAIC Company) 2006, *Evaluation of Neutron Absorber Materials Used for Criticality Control in Waste Packages* [DIRS 180664], CAL-DS0-NU-000007. Las Vegas, Nevada: Bechtel SAID Company. ACC: DOC20061009.0003.

SNL (Sandia National Laboratories) 2007, *Analysis of Dust Deliquescence for FEP Screening* [DIRS 181267], ANL-EBS-MD-000074, rev 01 ad 01. Las Vegas, Nevada: Sandia National Laboratories. ACC: DOC.20070911.0004.

SNL (Sandia National Laboratories) 2007, *General Corrosion and Localized Corrosion of the Drip Shield*[DIRS 180778], ANL-EBS-MD-000004 rev 02. Las Vegas, Nevada: Sandia National Laboratories. ACC: DOC.20070807.0004.

SNL (Sandia National Laboratories) 2007, *General and Localized Corrosion of the Waste Package Outer Barrier* [DIRS 178519], ANL-EBS-MD-000003 rev 03. Las Vegas, Nevada: Sandia National Laboratories. ACC: DOC.20070730.0003.

SNL (Sandia National Laboratories) 2007, *Stress Corrosion Cracking of Waste Package Outer Barrier and Drip Shield Materials* [DIRS 181953], ANL-EBS-MD-000005 rev 04. Las Vegas, Nevada: Sandia National Laboratories. ACC: DOC20070913.0001.

DISTRIBUTION

1	MS 0778	P.N. Swift	6783
2	MS 0888	D. G. Enos	1825
5	MS 0888	F. D. Wall	1823
2	MS 0776	K.B. Helean	6782
1	MS 0888	K.R. Zavadil	1825
1	MS 1399	S. A. Orrell	6800
1	MS 1399	C. L. Howard	6785
1	MS 1399	K. M. Knowles	6781
1	MS 1399	E. J. Bonano	6783
1	MS 0776	F.D. Hansen	6785
10	MS 1399	N. R. Brown	6781
1	MS 1399	D. Thomas	6785
1	MS 1399	K. Mon	6785
1	MS 1399	G. De	6785
1	MS 1399	G. Gordon	6785
1	MS 1399	M. Sutton	6780
3	MS 1399	Jim Blink	6780
1	MS 0899	Technical Library (Electronic Copy)	9536

Sandia National Laboratories